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Report No. AMXTH-TE-CR-86092

Contract DAAK 11-85-C-0007 (Task Order 4) Bench-Scale Investigation of Air Stripping of Volatile Organic Compounds (VOC's) From Soil

Technical Report

January 1987

Prepared for:
U.S. ARMY TOXIC AND HAZARDOUS MATERIALS AGENCY
Aberdeen Proving Ground (Edgewood Area), Maryland 21010

WESTON
Roy F. Weston, Inc.
West Chester
Pennsylvania

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report presents the results of a benchscale investigation which evaluated the role of aeration in thermal stripping of volatile organic compounds (VOC's) from soil.
The project included: Process equipment design, development of a test plan, bench scale investigation and evaluation of results.

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1. EXECUTIVE SUMMARY

Soils at several U.S. Army Materiel Command (AMC) installations have been contaminated with a variety of organic compounds as a result of past solvent handling practices. In many cases the contaminated soil has resulted in the degradation of underlying groundwater supplies.

In order to limit contaminant migration, the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) is investigating technologies to effectively treat the contaminated soil. One treatment alternative is low temperature thermal stripping of volatile organic compounds (VOC's) from soil. The concept of low temperature thermal stripping essentially couples two removal mechanisms:

(a) Removal by thermal volatization.
(b) Removal by aeration.

To determine the singular effect of these removal mechanisms, two separate studies were conducted at the Letterkenny Army Depot (LEAD), located in Chambersburg, Pennsylvania. A pilot study was conducted to evaluate removal by thermal volatization. During the pilot study, a thermal processor was used to heat and consequently dry the contaminated soil. The net effect of heating the soil was to evaporate volatile contaminants in the soil. In addition to the pilot study, a separate benchscale study was conducted to evaluate removal by aeration. The benchscale investigation was conducted simultaneously with the pilot investigation. A portion of the soils excavated for use in the pilot study were used in the benchscale investigation. This report presents the results of the benchscale study conducted during the period from 28 August 1985 to 13 September 1985.

The primary objective of the benchscale investigation was to determine the role of aeration in thermal stripping. Secondary objectives included the following:

(a) Determination of the impact of varying design parameters (i.e., inlet air pressure, operating temperature) on system performance (i.e., VOC removal efficiency).
(b) Evaluation of the feasibility for a pilot-scale demonstration of the air stripping concept.

Soils from the site of the two lagoons that were apparently used for the disposal of organic liquids were chosen for treatment. This selection was based on the type, variety, concentration, and volatile nature of the compounds found in this area. Two types of soil existed at this site: fill soil and native soil. A grain size analysis indicated that the fill material consisted of gravelly sands, and the native soil consisted of sandy clay/sandy silt.

For the benchscale application, an aeration unit was specially designed and fabricated. A shallow bed of contaminated soil was placed on top of the aeration surface. The unit allowed intimate contact between the air stream and contaminated soil. The net effect was to aerate the soil, thereby stripping the VOC's from the contaminated soil.

Four test runs were completed during the benchscale investigation. Two levels of inlet air pressure and, thus, two levels of inlet air temperature were evaluated to determine the effect on VOC removal efficiency: 3 pounds per square inch (psi) and 5 psi. The resulting inlet air temperatures were 144°F and 137°F for 3 psi and 148°F and 163°F for 5 psi. The discharge temperatures for each pressure are not the same because inlet air conditions (i.e., ambient temperature and moisture content) affect the outlet temperature and were different on each day of testing.

Based on review of the data associated with all test runs, the following conclusions are presented:

1. VOC removal efficiency is related to total VOC concentration in feed soils.

2. There is no apparent correlation between the soil bed temperature and VOC removal efficiency.

3. Inlet air temperature appears to be inversely related to VOC removal efficiency.

4. There is no apparent correlation between the moisture content in the inlet air and the VOC removal efficiency.

5. The greatest VOC removal occurs during evaporation of moisture from the soil.

6. Processed soil moisture content provides an indication of VOC removal efficiency and possibly processed soil VOC residuals.

7. Comparison of the VOC removal efficiencies associated with the aeration element and the thermal element (discussed in a separate report[1]) indicates that the role of aeration in thermal stripping is minimal. This conclusion applies to those conditions evaluated in this study (i.e., inlet air pressure, inlet air temperature, inlet air moisture content, ambient air temperature, and test duration).

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[1] Task 11. Pilot Investigation of Low Temperature Thermal Stripping of Volatile Organic Compounds (VOC's) From Soil, Report No. AMXTH-TE-CR-86074, June 1986.

2. INTRODUCTION

2.1 Background. Soils at several U.S. Army Materiel Command (AMC) installations have been contaminated with a variety of organic compounds as a result of past solvent handling practices. In many cases the contaminated soil has resulted in the degradation of underlying groundwater supplies.

In order to limit contaminant migration, the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) is investigating technologies to effectively treat the contaminated soil. One treatment alternative is low temperature thermal stripping of volatile organic compounds (VOC's) from soil. The concept of low temperature thermal stripping essentially couples two removal mechanisms:

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To determine the singular effect of these removal mechanisms, two separate studies were conducted at the Letterkenny Army Depot (LEAD), located in Chambersburg, Pennsylvania. A pilot study was conducted to evaluate removal by thermal volatization. During the pilot study, a thermal processor was used to heat and consequently dry the contaminated soil. The net effect of heating the soil was to evaporate volatile contaminants in the soil. In addition to the pilot study, a separate benchscale study was conducted to evaluate removal by aeration. The benchscale investigation was conducted simultaneously with the pilot investigation. A portion of the soils excavated for use in the pilot study were used in the benchscale investigation. This report presents the results of the benchscale study conducted during the period from 28 August 1985 to 13 September 1985.

2.2 Purpose of the report. The purpose of this report is to present the results and conclusions of a benchscale investigation that evaluated the concept of air stripping of VOC's from soil. A description of test conditions and process equipment is contained herein.

2.3 Objectives of the benchscale study. The primary objective of the benchscale investigation was to determine the role of aeration in thermal stripping. Secondary objectives included the following:

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[1] Task 11. Pilot Investigation of Low Temperature Thermal Stripping of Volatile Organic Compounds (VOC's) From Soil, Report No. AMXTH-TE-CR-86074, June 1986.

(a) Determination of the impact of varying design parameters (i.e., inlet air pressure, operating temperature) on system performance (i.e., VOC removal efficiency).
(b) Evaluation of the feasibility for a pilot-scale demonstration of the air stripping concept.

2.4 Report organization. The information contained in this report has been organized into 9 sections:

Section	Title
1	Executive Summary
2	Introduction
3	Test Site
4	Description of the Process Equipment
5	Experimental Variables
6	Sampling Techniques and Analytical Methods
7	Presentation of Data
8	Analysis of Results
9	Conclusions and Recommendations

The Appendices provide additional data and analyses:

Appendix	Title
A	Organic Waste Characteristics of Site Soils at LEAD (Determined During Preliminary Investigations)
B	Grain Size Gradation Curves Corresponding to Fill Soil and Native Soil
C	Analytical Methods
D	Supplemental Data

3. TEST SITE

3.1 Test site location and description. The benchscale investigation was conducted at the Letterkenny Army Depot (LEAD). LEAD, formerly known as Letterkenny Ordnance Depot, consists of 7,899 hectares (nearly 20,000 acres) of land situated in the south-central section of Pennsylvania in Franklin County, near the city of Chambersburg. A site location map for the installation is presented in Figure 3-1.

LEAD was established on 7 January 1942 with the mission of ammunition storage. The present expanded mission of LEAD includes the receipt, storage, inventory, maintenance, and demilitarization of ammunition; the overhaul, rebuilding, and testing of wheeled and tracked vehicles; and the issue and shipment of Class III chemicals and petroleum.[2] Some facility operations have included cleaning and stripping, plating, lubrication, demolition, chemical and petroleum transfer and storage, and washout/deactivation of ammunition.[3]

Soils excavated from Area K-1 were used in the benchscale investigation (as well as the pilot investigation discussed in Subsection 2.1). Area K-1 is one of seven potential hazardous waste disposal sites located in the East Patrol Road Disposal Area (EPRDA). EPRDA is located east of California Avenue, south and west of East Patrol Road, and north of Building 370. The location of Area K-1 is shown in Figure 3-2.

3.2 Waste characteristics. Previous efforts have identified and quantified the contaminants present in the site soils at LEAD.[4] In addition to VOC's, concentrations of asbestos, zinc, lead, copper, and cadmium have been found in Area K-1. However, since the benchscale study addressed VOC's only, other contaminants were not evaluated and will not be discussed.

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[2] USATHAMA Installation Assessment of Letterkenny Army Depot, January 1980.

[3] Battelle, Interim Report, Environmental Contamination Survey of Letterkenny Army Depot (LEAD), Part 1: Exploratory Phase, Draft, May 1982.

[4] Letterkenny Army Depot Remedial Investigation and Feasibility Study, Report No. DRXTH-AS-CR-83247, February 1984.

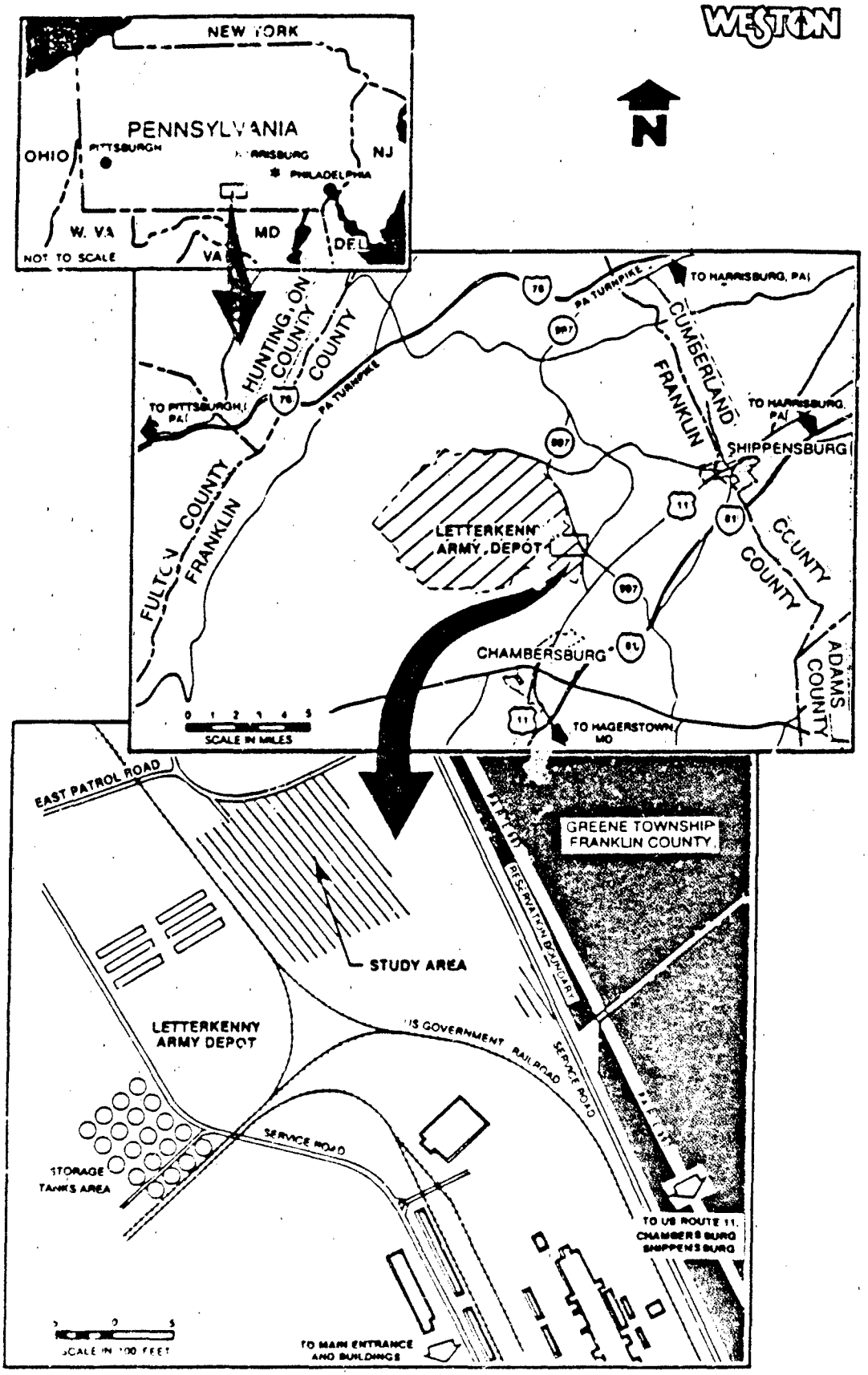

FIGURE 3-1 GENERAL LOCATION MAP OF THE STUDY AREA ON THE LETTERKENNY ARMY DEPOT, FRANKLIN COUNTY, PENNSYLVANIA

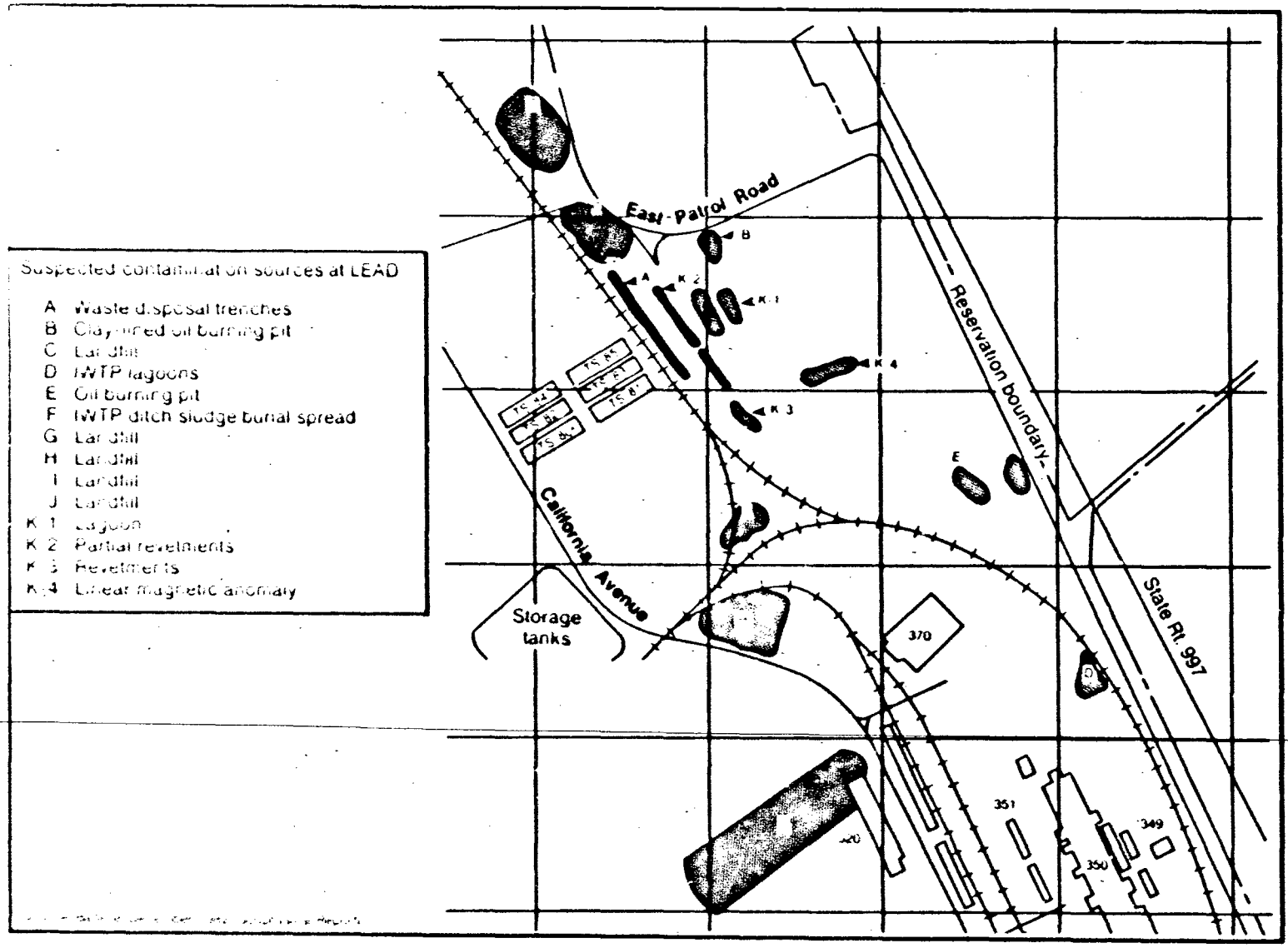

FIGURE 3-2 LOCATIONS OF POTENTIAL CONTAMINANT SOURCES EAST PATROL ROAD DISPOSAL AREA, LETTERKENNY ARMY DEPOT

Prior to the pilot study and benchscale investigation, a field sampling program was conducted on 10, 11, and 12 June 1985. During this program, soil sampling was conducted in Area K-1. Eleven boreholes were drilled to a depth of 10 feet. Five composite soil samples per borehole were collected at various depths. All soil samples were analyzed for those VOC's listed on the Hazardous Substance List (HSL). A list of the VOC's contained on the HSL, as well as their detection limits, is provided in Appendix A. A list of VOC's determined to have been present in Area K-1, along with their corresponding concentration range, is also contained in Appendix A. For convenience, the major compounds that were found to be present in Area K-1 are shown, along with maximum and average concentrations, in Table 3-1.

The pilot study was conducted simultaneously with the benchscale investigation and was completed in two phases: Phase 1 - 18 test runs; Phase 2 - 10 test runs. A summary of the VOC concentrations in the excavated soils used in Phase 1 and Phase 2 is included in Table 3-2. A detailed list of VOC concentrations for each test run is included in Appendix A.

3.3 Site/soil characteristics.

3.3.1 Site characteristics. Area K-1 is the site of two lagoons that were allegedly used for the disposal of organic liquids, as evidenced by the high concentrations of organic contaminants found in the soil. However, excavation operations indicated that a wide variety of miscellaneous debris was also deposited at this site. Typically, at a depth of approximately 3 to 5 feet an assortment of miscellaneous objects were unearthed (i.e., brake drums, wire, bolts, metal washers, bottles, shell casings, rubble, and trash).

3.3.2 Soil characteristics. The soil series for Area K-1 are classified as Urban Land. According to the Soil Conservation Service (SCS) of Franklin County, Pennsylvania, urban land is land that is so altered that identification of soils is not feasible. This series generally consists of nearly level to sloping land that has been affected by urban development. Included in this unit are soils that have been cut and filled with earth and trash material.

TABLE 3-1. CONCENTRATION RANGE OF VOC'S DETERMINED TO BE PRESENT IN AREA K-1 (BASED ON TESTING PERFORMED ON 10, 11, 12 JUNE 1985)

Volatile organic compound	Average concentration (ppm)	Maximum concentration (ppm)
1,2-Trans Dichloroethylene	115	>1,300
Trichloroethylene	222	>3,500
Tetrachloroethylene	95	>3,800
Xylene	7	47
Other VOC's (i.e., Chlorobenzene, Ethylbenzene, Methylene chloride, Toluene, Vinyl chloride, C_{10}-allyl Benzene, Dichlorobenzene, methyl ethyl benzene, n-propylbenzene, Trimethyl benzene)	7	600

ppm = parts per million

TABLE 3-2. VOC CONCENTRATIONS IN EXCAVATED SOILS FROM PHASE 1 AND PHASE 2 OF THE PILOT INVESTIGATION

Volatile organic compound	Average concentration (ppm)	Maximum concentration (ppm)
Phase 1		
1,2-Trans Dichloroethylene	252	1,200
Trichloroethylene	2,729	20,000
Tetrachloroethylene	745	4,800
Xylene	86	460
Other VOC's	38	270
Phase 2		
1,2-Trans Dichloroethylene	18	74
Trichloroethylene	>146	>390
Tetrachloroethylene	>94	>260
Xylene	>62	>7,190
Other VOC's	11	35

Excavations in Area K-1 indicated that a gravelly sandy silt fill covered the surface to an approximate depth of 2 feet. From 2 to 5 feet below ground surface, miscellaneous fill material consisting of gray silty clay with sand, gravel, black ash, and metallic debris was encountered. Native soils varying from orange brown, sandy, gravelly plastic clays to slightly plastic clayey silts were generally observed between 5 to 7 feet. In addition, a perched water table was occasionally observed at the interface of the native soil and fill.

4. DESCRIPTION OF THE PROCESS EQUIPMENT.

4.1 Aeration unit. The aeration unit evaluated in the benchscale study is used industrially to aid in the withdrawal of dry, relatively free flowing material from storage bins and silos. The unit supplies a low-pressure diffused air surface which fluidizes a thin layer of material, thereby promoting flow by gravity.

For the benchscale application, a shallow bed of contaminated soil was placed on top of the aeration surface. A constant flow rate of air was diffused by the surface. The unit allowed intimate contact between the air stream and contaminated soil. The net effect was to aerate the soil, thereby stripping the VOC's from the contaminated soil.

An illustration of the aeration unit is presented in Figure 4-1. The heart of the aeration unit is an aluminum oxide porous plate housed in a cast iron casing. The porous plate and housing measures 15-1/2 inches long by 15-1/2 inches wide by 3 inches thick and results in approximately 150 square inches of surface area. The casing is flange mounted on the underside of an open-bottom container. The container walls are approximately 2 feet high and constructed of stainless steel on three sides and safety glass on the fourth side (to view the soil during treatment). The container wall constructed of safety glass is removable for access to the unit (loading, sampling, etc). The "door" is attached with a series of C-clamps. Originally the door was to be bolted on; however, the process of removing the bolts was too time-consuming during soil sampling. The top of the container has a pitched stainless steel cover with a 2-inch diameter air discharge pipe.

The diffuser plate casing was fitted with a standard pipe connection (3/4-inch diameter) to admit process air. The unit was designed to accommodate 15 dry standard cubic feet per minute (dscfm) of air at a pressure of up to 5 pounds per square inch (psi). A low pressure rotary lobe blower supplied the process air. The air stream was diffused by the porous plate, passed through a stationary bed of soil (approximately 1-1/2 inches high), exited the unit through the air discharge line, and, finally, was directed to an afterburner for conversion of the VOC's to hydrochloric acid, carbon dioxide, and water vapor.

The afterburner (designed and fabricated primarily for use in the pilot study that was being conducted simultaneously) operated at a minimum temperature of 1,000°C (1,832°F) and had a residence time of greater than two seconds. The afterburner was propane-fired, using a North American burner rated at 1.5 million British thermal units (Btu) per hour. The afterburner operated in conjunction with a refractory-lined stack that was 18 inches in diameter and 20 feet high.

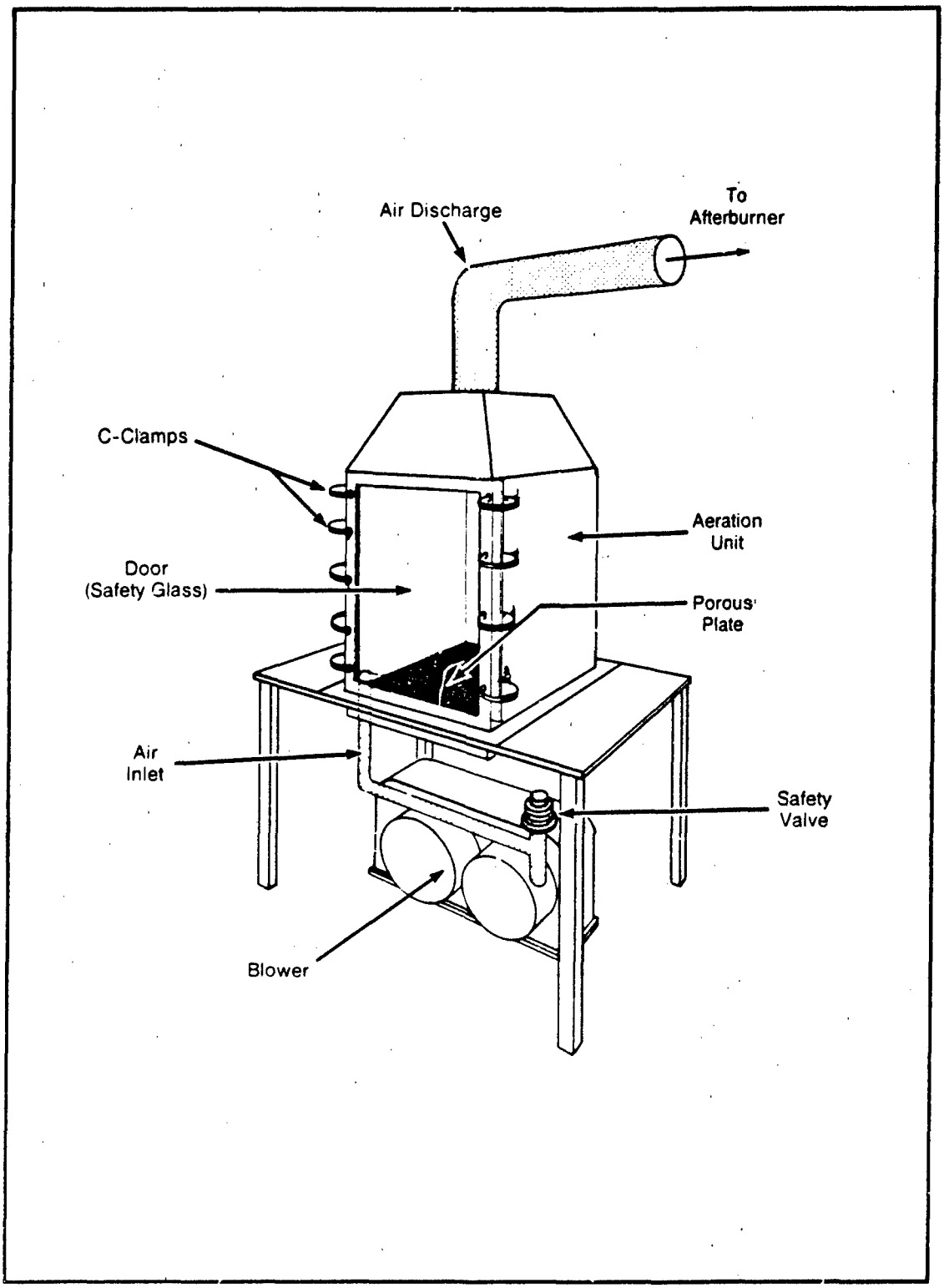

FIGURE 4-1 SCHEMATIC OF AIR STRIPPING PROCESS EQUIPMENT

5. EXPERIMENTAL VARIABLES

The variables of the benchscale study were classified as follows:

(a) Independent variables - Those variables impractical to control and allowed to vary randomly throughout the tests. No attempts were made to modify or control independent variables.
(b) Control variables - Those variables with values selected and maintained during test operations.
(c) Response variables - Those variables with values that were a function of the selected operating conditions.

Table 5-1 provides a summary of test variables associated with the aeration unit. A brief discussion of the variables is included in the following subsections.

5.1 Independent variables. As shown in Table 5-1, there were two independent variables associated with the benchscale study. These independent variables were the feed soil composition/conditions (i.e., VOC concentrations, moisture content, and temperature) and the inlet air composition/conditions (i.e., VOC concentrations, moisture content, and ambient temperature).

5.1.1 Feed soil composition/conditions. One goal of the benchscale study was to determine the capability of the air stripping equipment to treat actual contaminated soils. Therefore, the composition/conditions of the soils in Area K-1 were not altered prior to being introduced to the unic. The VOC concentration and moisture content of feed soils were a function of the location and depth of soils excavated for treatment. The temperature of the feed soils depended on ambient conditions at the time of the test (soils were stored in sealed metal containers on the processing pad).

5.1.2 Inlet air composition/conditions. Various activities involving the contaminated soils (i.e., sampling, excavation) took place during the benchscale study. Therefore, the potential existed for trace concentrations of fugitive VOC's to be present in the influent air stream. No attempts were made to modify the inlet VOC concentration, although it was monitored (as discussed in subsection 6.1.2.4). The moisture content and temperature of the air stream were a function of ambient conditions.

TABLE 5-1. SUMMARY OF TEST VARIABLES FOR THE AERATION UNIT

A. INDEPENDENT VARIABLES

Feed Soil Composition/Conditions

- VOC Concentrations
- Moisture Content
- Temperature

Air Inlet Composition/Conditions

- VOC Concentrations
- Moisture Content
- Ambient Temperature

B. CONTROL VARIABLES

Held Constant Throughout Testing Program

- Feed Soil Volume
- Air Flow Rate
- Soil Residence Time

Held Constant At Various Levels

- Air Pressure at Inlet

C. RESPONSE VARIABLES MEASURED

Soil Composition/Conditions

- VOC Concentrations (during and after batch test)
- Moisture Content (during and after batch test)
- Temperature (during batch test)
- Mass (before and after batch test)

Air Composition/Conditions

- VOC Concentrations (discharge air)
- Moisture Content (discharge air)
- Temperature (inlet and discharge air)
- Pressure (discharge)

5.2 Control variables. As shown on Table 5-1, there were three variables held constant at all levels (i.e., feed soil volume, air flow rate, and soil residence time) and one variable held constant at various levels (i.e., inlet air pressure). A schedule of test runs, as well as control variables, is shown in Table 5-2.

5.2.1 Control variables held constant at all levels. A constant volume of soil (approximately 4.5 liters) was treated during each batch test run. Soil was manually delumped and rocks and oversized items were removed. The constant volume resulted in approximately 10 pounds of contaminated soil. The approximate bed height was 1.5 inches.

A constant volume, low pressure rotary lobe blower maintained an air flow rate of approximately 15 dry standard cubic feet per minute (dscfm) during each test run.

The soil residence time was approximately 260 minutes for each test run, but varied slightly.

5.2.2 Control variables held constant at various levels. The pressure of the inlet air stream was evaluated at two levels: 3 psi and 5 psi. The major reason for varying pressure was to evaluate two levels of inlet air temperature (as temperature is directly related to blower discharge pressure due to the associated heat of compression).

5.3 Response variables measured.

5.3.1 Soil composition/conditions. Treated soils were sampled at the end of Test Runs 1 and 2 to determine the overall VOC removal efficiency. In addition, to determine the VOC removal trend (over time), the aeration unit was opened and soils were sampled at discrete intervals during Test Runs 3 and 4.

The temperature of the soil bed, dependent on the temperature of the inlet air stream, was monitored at discrete intervals over the duration of each test run.

The mass of the soil changed over the duration of the test run as moisture in the soil evaporated. To determine the approximate amount of moisture that exited the unit as water vapor, the mass of the feed and processed soils were measured for each test run.

TABLE 5-2. SCHEDULE OF TEST RUNS FOR THE AERATION UNIT

Test run	Test run date	Volume of soil treated (liters)	Target air flow rate (dscfm)	Target soil residence time (minutes)	Target inlet air pressure (psi)
1	8/29/85	4.5	15	260	5
2	9/6/85	4.5	15	260	3
3	9/12/85	4.5	15	260	5
4	9/13/85	4.5	15	260	3

5.3.2 Air composition/conditions. The VOC concentration in the discharge air was monitored over the duration of each test run to determine the VOC removal trend.

The moisture contents of the inlet air stream and discharge air stream were monitored at the beginning and end of each test run.

The temperature of the inlet air stream was a function of the blower discharge pressure (due to the heat of compression). To determine the air temperatures corresponding to selected discharge pressures, the temperature of the inlet air stream was monitored at discrete intervals during each test run.

The pressure of the air stream discharging the aeration unit was monitored at discrete intervals during each test run to determine the pressure drop over the unit.

6. SAMPLING TECHNIQUES AND ANALYTICAL METHODS

A brief discussion of the techniques used to sample the soil and air streams, as well as the laboratory methods used to analyze the samples, is contained in the following subsections. An instrumentation diagram showing the location of measuring devices is included in Figure 6-1.

6.1 Field sampling techniques.

6.1.1 Soil sampling techniques. A list of the soil parameters that were monitored and/or sampled for analysis is contained in Table 6-1. As shown, four parameters were monitored and/or sampled for in the field: those VOC's listed on the HSL (Appendix A), moisture content, temperature, and mass.

6.1.1.1 VOC's. A 40-milliliter volatile organic analysis (VOA) vial was filled with feed soil, soil at intermediate stages of treatment (only during Test Runs 3 and 4), and treated soils for analysis of those VOC's on the HSL. The feed soil was sampled after it was manually delumped and placed in the aeration unit. The soil bed was sampled at various locations and depths to obtain a sample that was thought to be representative. No attempt was made to minimize VOC losses during delumping activities or placement into the aeration unit. Since the feed soil sample was not collected until after these activities were completed, the VOC concentrations in the samples should be representative of actual conditions at the beginning of the test.

When soils were sampled during the test run (Test Runs 3 and 4), the following sequence of events occurred:

1. The blower was shut off.
2. The C-clamps on the front door were removed.
3. The front door was removed.
4. VOA bottles were filled with soil.
5. The front door and C-clamps were replaced.
6. The blower was turned on and the test run resumed.

The entire sampling operation took about five to 10 minutes. No attempt was made to minimize VOC losses during intermediate sampling activities. It was thought that the amount of VOC's lost during sampling would be minimal when compared to those VOC's driven off during operation of the unit (i.e., 15 dry standard cubic feet per minute at a minimum temperature of 137°F). The soil samples were stored on ice until delivery to the WESTON laboratory.

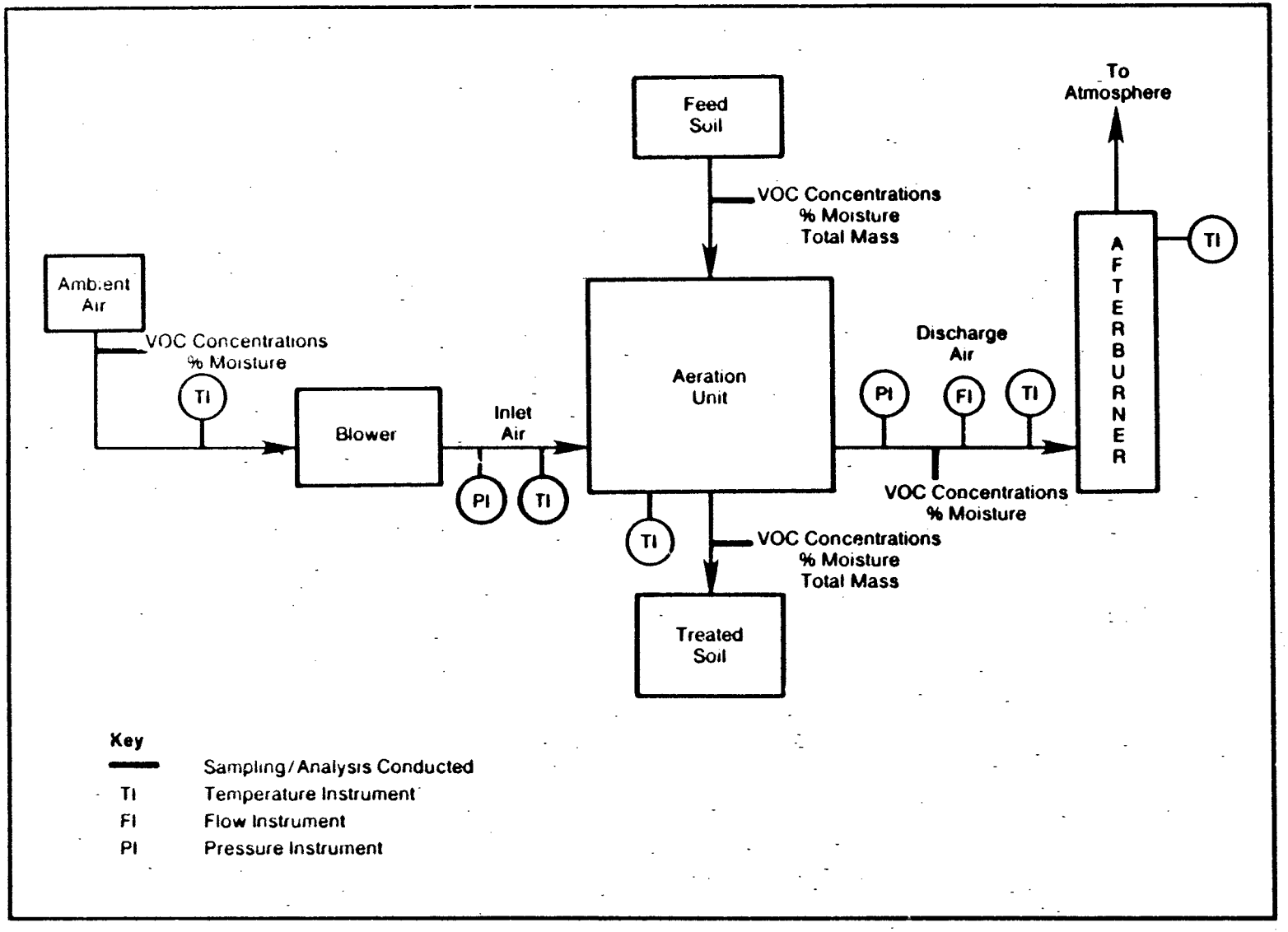

FIGURE 6-1 AERATION UNIT INSTRUMENTATION AND SAMPLING/ANALYSIS

TABLE 6-1. PARAMETERS MONITORED AND/OR SAMPLED FOR IN SOILS

1.	VOC's	Feed Soil Soil during treatment (Test Runs 3 and 4 only) Treated Soil
2.	Moisture Content	Feed Soil Soil during treatment (Test Runs 3 and 4 only) Treated Soil
3.	Temperature	Feed Soil Soil during treatment (All test runs)
4.	Mass	Feed Soil Treated Soil

6.1.1.2 Moisture content. A 40-milliliter VOA vial was filled with feed soil, soil being treated (during Test Runs 3 and 4) and treated soils. The soil samples were stored on ice until delivery to the WESTON laboratory for analysis.

6.1.1.3 Temperature. The temperature of the soil was monitored using a chromel-alumel thermocouple. A hole was drilled in the aeration unit wall and the thermocouple was inserted into the soil bed. The thermocouple was fully embedded in the soil and was not exposed to the air or porous plate. The thermocouple was wired to a multipoint calibrated digital pyrometer for accurate reading of temperature. The soil bed temperature was monitored and recorded at 5-minute intervals over the entire duration of the test.

6.1.1.4 Mass. As discussed in Subsection 5.2.1, a constant volume of soil (approximately 4.5 liters) was treated during each batch test run. An aluminum cake pan was used to measure the soil volume. A scale (accurate to $\pm$1 pound) was used to weigh the soil and cake pan. The weight of the empty cake pan was then subtracted to determine the soil mass. Soils were weighed before and after each batch test run.

6.1.2 Air sampling techniques. A list of the parameters that were monitored and/or sampled for in the air stream is contained in Table 6-2. As shown, five parameters were monitored and/or sampled for in the field: VOC's, moisture content, temperature, flow rate, and pressure. A brief discussion of the air sampling techniques is contained in the following subsections.

6.1.2.1 VOC's. Total VOC's in the aeration unit outlet were monitored by a continuous emissions monitoring (CEM) system during each test run. Gross VOC concentrations were monitored using an AID Model 590 volatile organics monitor/GC (photoionization detector with 10.0 electron-volt lamp). Tygon tubing connected the sample test port in the discharge line to the inlet port on the portable field instrument.

The CEM system measured gross VOC concentrations in the linear range from 1 to 600 ppm (by volume, dry basis) relative to the calibration gas (benzene). The total VOC concentrations were recorded at 5-minute intervals during each test run.

6.1.2.2 Moisture content. The moisture content of the inlet and outlet air streams was monitored at the beginning and end of each test run. The moisture content of the aeration unit inlet (blower discharge), assumed to be the same as ambient air, was measured using a sling psychrometer and associated psychrometric chart.

TABLE 6-2. PARAMETERS MONITORED AND/OR SAMPLED FOR IN THE AIR STREAM

1.	VOC's	Ambient Air Discharge Air
2.	Moisture Content	Ambient Air Discharge Air
3.	Temperature	Ambient Air Inlet Air Discharge Air
4.	Flow Rate	Discharge Air
5.	Pressure	Inlet Air Discharge Air

The moisture content of the aeration unit outlet air was determined using the wet bulb temperature (measured by inserting a chromel-alumel thermocouple with wet sock into the outlet line), the dry bulb temperature (measured by inserting a chromel-alumel thermocouple into the outlet line), and a psychrometric chart.

Moisture contents were monitored and recorded at the beginning and end of each test run.

6.1.2.3 Temperature. The temperature of the air stream was monitored at three locations: ambient air, aeration unit inlet (blower discharge), and aeration unit outlet.

The temperature of the ambient air was monitored using a mercury thermometer. Ambient air was monitored and recorded every 30 minutes.

The temperature of the inlet air stream increased with the blower discharge pressure (due to heat of compression). The corresponding temperature of the aeration unit inlet was monitored using a bimetal thermometer inserted into the blower discharge line. The temperature of the inlet stream was monitored and recorded every five minutes.

A bimetal thermometer was also inserted into the aeration unit outlet stream to monitor temperature. The temperature was monitored and recorded every five minutes during each test run.

6.1.2.4 Flow rate. The flow rate of air into the aeration unit was assumed to be the same as the flow rate of air out of the unit. Standard pitot tubes were used in conjunction with inclined manometers to measure the flow in the outlet stream. The flow rate was monitored and recorded at the beginning and end of each test run.

6.1.2.5 Pressure. The pressure on the blower was controlled by adjusting the weight of washers on a 1-inch diameter safety relief valve. As metal washers were removed from the valve, the corresponding blower discharge pressure decreased.

The pressure was originally to be monitored using a bourdon C-tube pressure gauge. However, two gauges purchased in the field both malfunctioned; therefore, the blower discharge pressure was estimated, as discussed below.

The washers were weighed using a balance scale (accurate to ±1 gram). The resulting mass was 1,795 grams. This weight was converted to pressure using the following equation:

$$\text{Pressure } \left(\frac{\text{pounds}}{\text{inch}^2}\right) = \frac{\text{Weight of washers (pounds)}}{\text{Area of safety relief valve (inch}^2\text{)}}$$

$$= \frac{(1{,}795 \text{ grams}) \times (1 \text{ pound}/454 \text{ grams})}{(\pi/4) \times (1 \text{ inch})^2}$$

$$\approx 5.0 \text{ psi}$$

Two levels of discharge pressure were evaluated: 5 psi and 3 psi. To determine the weight of washers that must be removed from the relief valve to maintain 3 psi, the following equation was used:

$$3 \frac{\text{pounds}}{\text{inch}^2} = \frac{\text{weight of washers (grams)} \times (1 \text{ pound}/454 \text{ grams})}{(\pi/4) \times (1 \text{ inch})^2}$$

Weight of washers = 1,070 grams

This weight corresponded to 8 washers (actual weight of washers was 1,090 grams, resulting in an actual discharge pressure of 3.06 psi).

The pressure in the aeration unit outlet stream was measured using a water column pressure gauge. The differential pressure between the discharge air and atmospheric air was monitored and recorded every five minutes during each test run.

6.2 Analytical techniques. All soil samples were stored on ice until delivery to the WESTON laboratory. Upon arrival at the laboratory, all chain-of-custody forms were signed and samples were recorded in a bound logbook. All sample containers were maintained at 4°C until analyzed. No sample was retained longer than allowable holding times (i.e., 14 days). Analytical parameters and methods are listed in Table 6-3. Detailed descriptions of the analytical methods are contained in Appendix C. A brief discussion is contained in the following subsections.

TABLE 6-3. ANALYTICAL PARAMETERS AND METHODOLOGIES

Parameter	Method[1]
A. VOC's in soil.	EPA Contract Laboratory Protocol (CLP) for GC/MS Analysis of Purgeable Organics in Soils and Sediments.
B. Moisture Content of Soil.	Standard Method 209G.

[1]Descriptions of the methods are provided in Appendix C.

6.2.1 VOC's in soil. Volatile organics in soil samples were analyzed using the EPA Contract Laboratory Protocol (CLP) method for "GC/MS Analysis of Purgeable Organics in Soils and Sediments." Low level samples (i.e., those containing 5 to 2000 parts per billion (ppb)) were by the "low level protocol" in which an inert gas was bubbled through a mixture of a 0.005 to 5 gram sample and reagent water contained in a purging chamber at elevated temperatures. The purgeables were efficiently transferred from the aqueous phase to the vapor phase. The vapor was swept through a sorbent column where the purgeables were trapped. After purging was completed, the sorbent column was heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph was temperature programmed to separate the purgeables which were then detected with a mass spectrometer.

Samples containing higher levels (i.e., greater than 2000 parts per billion (ppb)) of purgeable organics were analyzed using the "medium level protocol." In this procedure a measured amount of soil was extracted with methanol. A portion (5 to 100 milliliters) of the methanol extract was diluted to 5 milliliters with reagent water. An inert gas was bubbled through this solution at ambient temperature in a specifically designed purging chamber. The purgeables were effectively transferred from the aqueous phase to the vapor phase. The vapor was swept through a sorbent column where the purgeables were trapped. After purging was completed, the sorbent column was heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph was temperature programmed to separate the purgeables which were then detected with a mass spectrometer as described in the CLP methods for "GC/MS Analysis of Purgeable Organics in Soils and Sediments," provided in Appendix C.

The calibration and quality control measures taken by the analytical laboratory are discussed in the following subsections.

6.2.1.1 Calibration. Mass spectrometers are tuned on a daily basis to manufacturer's specifications with FC-43. In addition, once per shift, these instruments are tuned with decafluorotriphenylphosphine (DFTPP) or 4-bromo-fluorobenzene (BFB) for semivolatiles or volatiles, respectively. Ion abundances will be within the windows dictated by the specific program requirements. Once an instrument has been tuned, initial calibration curves for analytes (appropriate to the analyses to be performed) are generated for at least three solutions containing known concentrations of authentic standards of compounds of concern. The calibration curve will bracket the anticipated working range of analyses.

Calibration data, to include the correlation coefficient, will be entered into laboratory notebooks to maintain a permanent record of instrument calibrations.

6.2.1.2 Quality Control. During each operating shift, a midpoint calibration standard is analyzed to verify that the instrument responses are still within the initial calibration determinations. The calibration check compounds will be those analytes used in the EPA Contract Laboratory Program's multicomponent analyses (e.g., priority pollutants and hazardous substances list) with the exception that benzene is used in place of vinyl chloride (volatiles) and di-n-octyl phthalate is deleted from the semivolatile list.

The response factor drift (percent RSD) will be calculated and recorded. If significant (>30 percent) response factor drift is observed, appropriate corrective actions will be taken to restore confidence in the instrumental measurements.

All GC/MS analyses will include analyses of a method blank in each lot of samples. In addition, appropriate surrogate compounds specified in EPA methods will be spiked into each sample. Recoveries from method spikes and surrogate compounds are calculated and recorded. All extractable analyses are accompanied by method spike/method spike duplicate data.

Duplicate samples will be analyzed for analytical lots of 20 or more.

Audit samples will be analyzed periodically to compare and verify laboratory performance against standards prepared by outside sources.

6.2.2 Moisture content in soil. The moisture content of soil was determined using Standard Method 209G. A copy of the method is provided in Appendix C. As a quality control measure, one laboratory blank and one replicate per batch (i.e., maximum of 20 samples)were also analyzed.

7. PRESENTATION OF DATA

7.1 Soil. Summaries of pertinent data corresponding to the soil medium for Test Runs 1, 2, 3, and 4 are included in Tables 7-1, 7-2, 7-3, and 7-4, respectively. Note that the detection limits for the feed soil and processed soil are different. This is because the detection limit depended on three factors:

1. the dilution factor,
2. the exact mass of soil weighed for analysis, and
3. the percent of moisture in the soil.

These three factors were different for each soil sample. The factor that had the greatest impact on detection limit was the dilution factor. The procedure for dilution is as follows:

1. Weigh mass of soil (target mass is recommended by analytical method).
2. Conduct analysis on soil, ensuring that the concentrations of target compounds are within the calibration range.
3. If the target compounds are not within the calibration range, use a lesser amount of soil than that used initially (i.e., a higher dilution factor and thus higher detection limit).

Also, note that some contaminant levels are estimated levels. In these cases, the mass spectral data indicated that the compound of concern was present, but the result was less than the specified detection limit but greater than zero. Estimations were made using the peak height and response factor.

To illustrate the trend of VOC removal, the total VOC concentrations in soils sampled during Test Runs 3 and 4 are shown as a function of time in Figures 7-1 and 7-2, respectively.

A detailed list of soil bed temperatures is shown as a function of time in Table D-1 in Appendix D.

7.2 Air. A summary of pertinent data corresponding to the air stream is shown in Table 7-5.

To evaluate the trend of VOC removal a detailed list of the total VOC concentration (as ppm by volume) in the discharge air stream is shown for each test run in Table D-2 in Appendix D. For illustration, the VOC removal trend (converted to pounds per hour) is shown graphically for each test run in Figures 7-3 through 7-6. Note that the removal trend is similar for each

test run; however, the ordinate on each figure is different. Therefore, the figures are not directly comparable (i.e., initial concentration for Test Run 4 is approximately 0.045 lb/hr, whereas initial concentration for Test Run 2 is approximately 0.002 lb/hr).

A detailed summary of inlet and outlet air temperatures is included in Table D-3 in Appendix D.

TABLE 7-1. SUMMARY OF MAJOR TEST VARIABLES IN SOIL TEST RUN 1

Conditions: Inlet Pressure - 5 psi
Residence Time - 230 minutes
Average Inlet Air Temperature - 163°F

	Feed soil	Treated soil	Removal efficiency (percent)
A. VOC Concentrations (ug/kg)			
1,2-Trans Dichloroethylene	33*	11**	67
Trichloroethylene	19*	43**	-126
Tetrachloroethylene	19*	6**	68
Xylene	490	23**	95
Other VOC's	86*	206	-140
Total VOC's	647	289	55
B. Moisture Content (Percent by weight)	17.8	0.6	97
C. Mass (pounds)	10	8	20

* Estimated value - detection limit was 120 ug/kg.
** Estimated value - detection limit was 50 ug/kg.

TABLE 7-2. SUMMARY OF MAJOR TEST VARIABLES IN SOIL TEST RUN 2

Conditions: Inlet Pressure - 3 psi
Residence Time - 245 minutes
Average Inlet Air Temperature - 144°F

	Feed soil	Treated soil	Removal efficiency (percent)
A. VOC Concentrations (ug/kg)			
1,2-Trans Dichloroethylene	ND	ND	---
Trichloroethylene	ND	9*	---
Tetrachloroethylene	ND	ND	---
Xylene	1,500	340	77
Other VOC's	38	109	-187
Total VOC's	1,538	458	70
B. Moisture Content (Percent by weight)	11.9	8.7	27
C. Mass (pounds)	11	9	18

ND - Not Detected
* Estimated value - detection limit was 50 ug/kg.
--- Not Applicable

TABLE 7-3. SUMMARY OF MAJOR TEST VARIABLES IN SOIL
TEST RUN 3

Conditions: Inlet Pressure - 5 psi
Residence Time - 285 minutes
Average Inlet Air Temperature - 148°F

	Feed Soil	Intermittent Soil Sample 1 (68 minutes)	Intermittent Soil Sample 2 (136 minutes)	Intermittent Soil Sample 3 (204 minutes)	Treated Soil	Overall Removal Efficiency (percent)
A. VOC Concentrations (ug/kg)						
1,2-Trans Dichloroethylene	98,000	26,000	15,000	17,000	18,000	82
Trichloroethylene	125,000	>260,000	39,000	35,000	35,000	72
Tetrachloroethylene	57,000	65,000	5,900	3,000	2,500	96
Xylene	8,200	4,800	230*	300**	330***	96
Other VOC's	3,740	2,092	232*	65**	1,108	70
Total VOC's	291,940	>357,892	60,362	55,365	56,938	81
B. Moisture Content (Percent by weight)	17.6	11.5	<0.10	<0.10	0.5	97
C. Mass (pounds)	10	NM	NM	NM	8	20

NM - Not Measured
* Estimated value - detection limit was 350 ug/kg.
** Estimated value - detection limit was 400 ug/kg.
*** Estimated value - detection limit was 850 ug/kg.

TABLE 7-4. SUMMARY OF MAJOR TEST VARIABLES IN SOIL
TEST RUN 4

Conditions: Inlet Pressure - 3 psi
Residence Time - 285 minutes
Average Inlet Air Temperature - 137°F

	Feed Soil	Intermittent Soil Sample 1 (68.5 minutes)	Intermittent Soil Sample 2 (136 minutes)	Intermittent Soil Sample 3 (204 minutes)	Treated Soil	Overall Removal Efficiency (percent)
A. VOC Concentrations (ug/kg)						
1,2-Trans Dichloroethylene	265,000	105,000	23,000	15,000	22,000	92
Trichloroethylene	1,420,000	1,350,000	131,000	62,000	104,000	93
Tetrachloroethylene	495,000	450,000	57,000	14,000	28,500	94
Xylene	56,500	24,000	6,100	1,300	1,300	98
Other VOC's	19,600	7,750*	3,540	1,310**	2,236***	89
Total VOC's	2,256,100	1,936,750	220,640	93,610	158,036	93
B. Moisture Content (Percent by weight)	18.8	12.6	3.2	4.4	0.7	96
C. Mass (pounds)	10	NM	NM	NM	9	10

NM - Not Measured
* Estimated value - detection limit was 3,000 ug/kg.
** Estimated value - detection limit was 1,200 ug/kg.
*** Estimated value - detection limit was 570 ug/kg.

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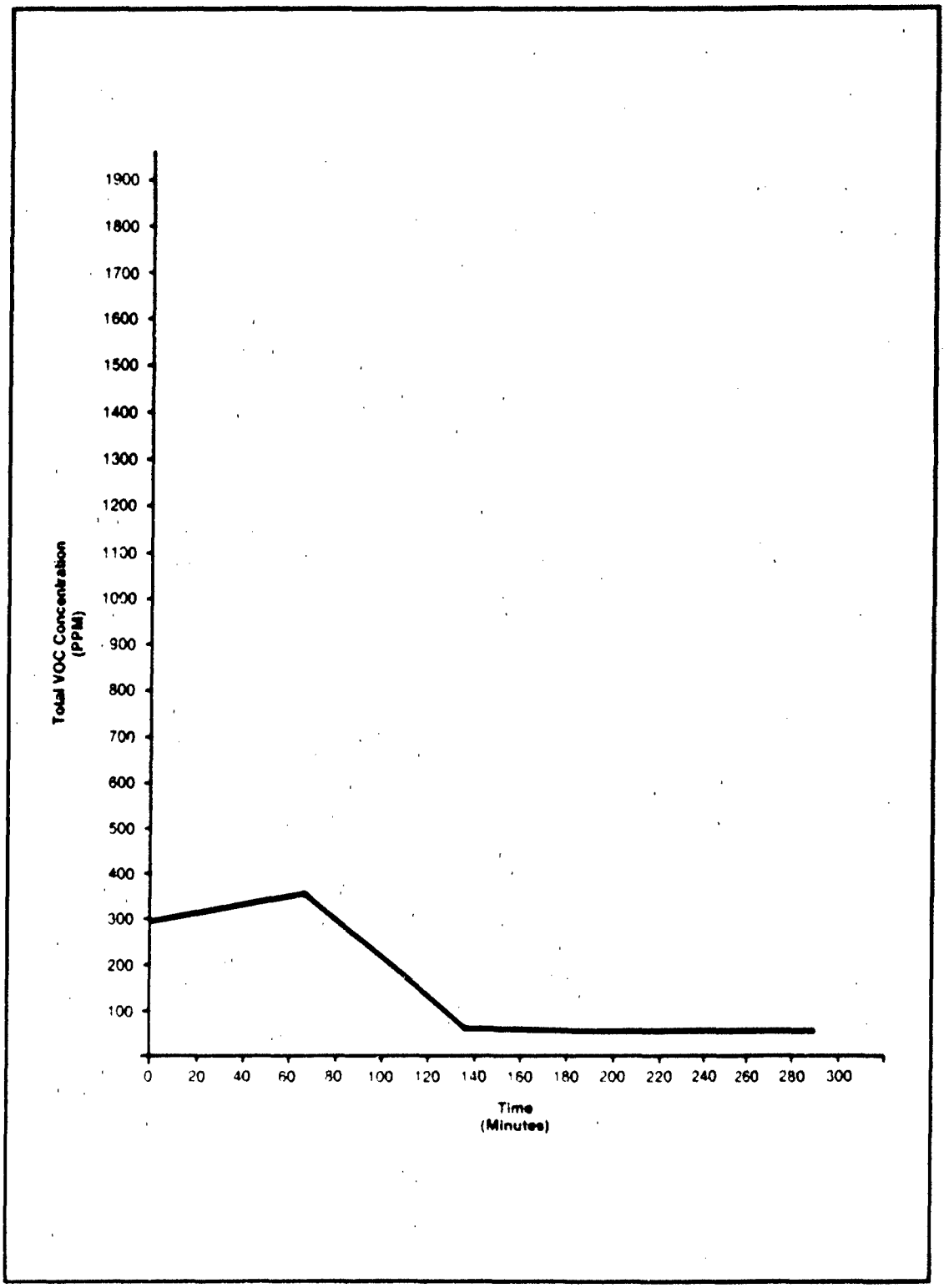

FIGURE 7-1 TOTAL VOC REMOVAL: TEST RUN 3

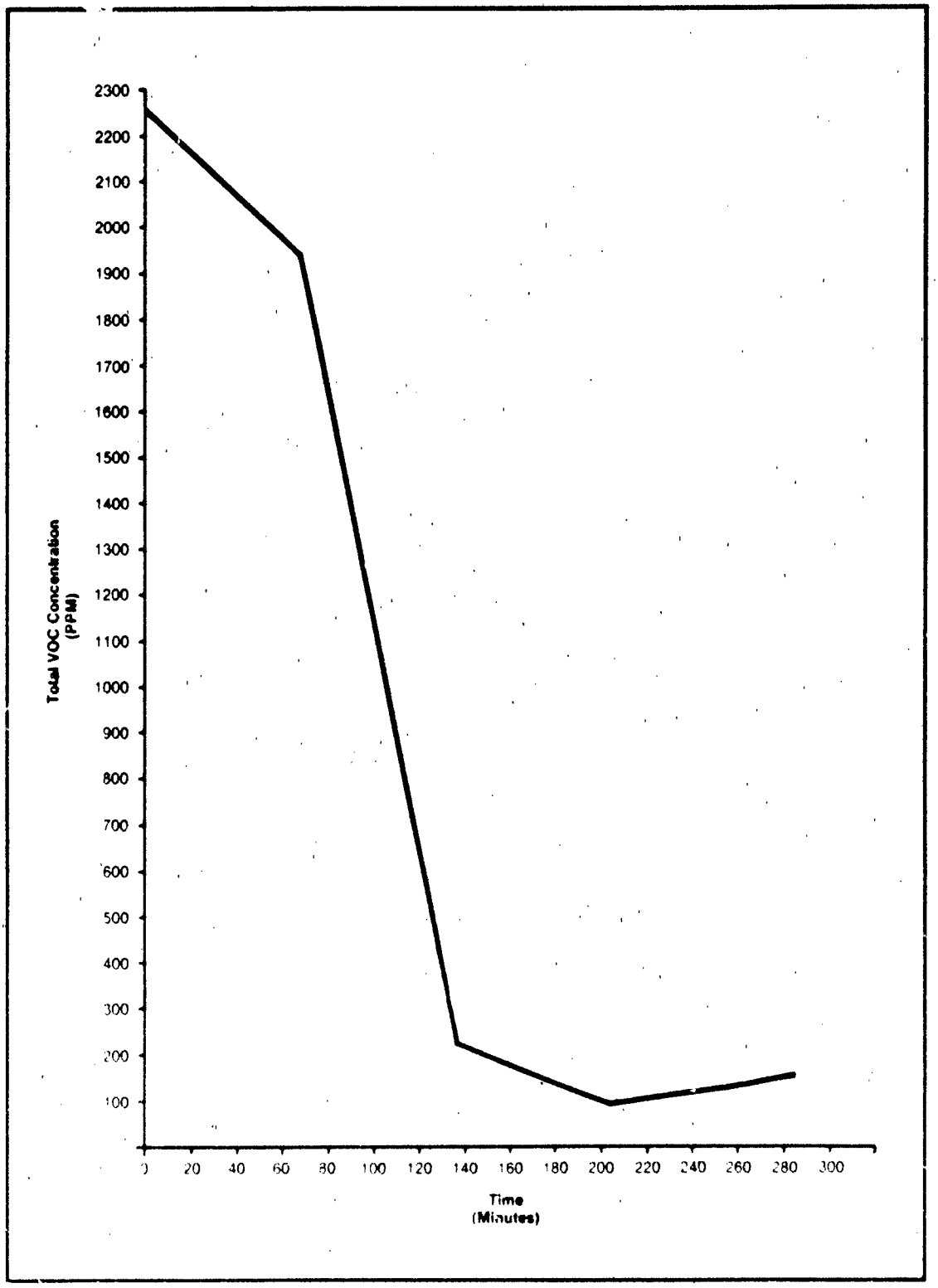

FIGURE 7-2 TOTAL VOC REMOVAL: TEST RUN 4

TABLE 7-5. SUMMARY OF MAJOR TEST VARIABLES IN AIR

	Test Run 1		Test Run 2		Test Run 3		Test run 4	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
A. Pressure	5	0.005	3	0.005	5	0.005	3	0.005
B. Total VOC's (ppm/volume as benzene)	<1	*	<1	**	<1	***	<1	****
C. Moisture Content (Percent by weight)	1.90	2.40	2.20	2.30	0.80	2.30	1.00	2.30
D. Flow Rate (dscfm)	NM	11.16	NM	11.11	NM	10.86	NM	11.45

NM - Not Measured
* See Figure 7-3
** See Figure 7-4
*** See Figure 7-5
**** See Figure 7-6

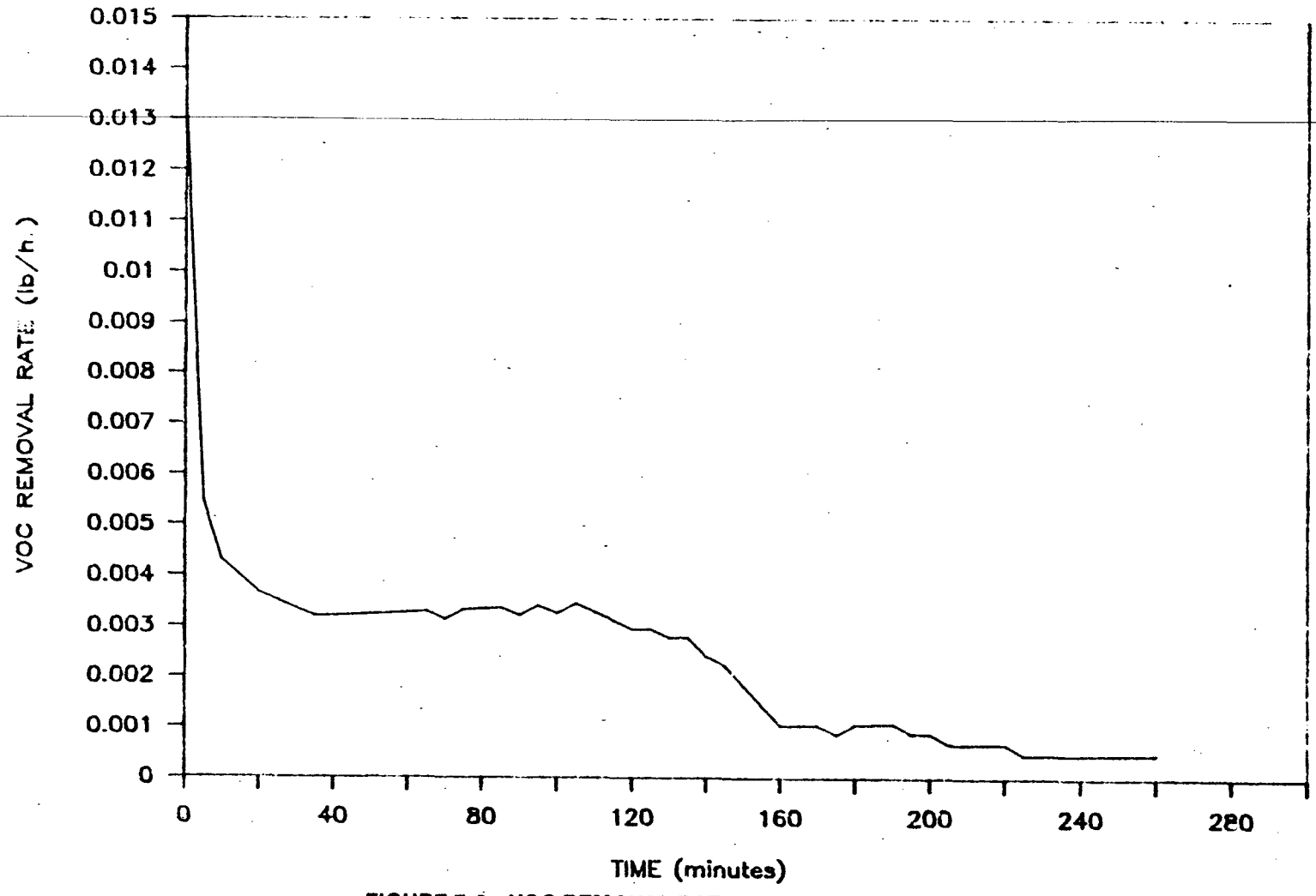

FIGURE 7-3 VOC REMOVAL RATE IN THE DISCHARGE AIR STREAM - TEST RUN 1

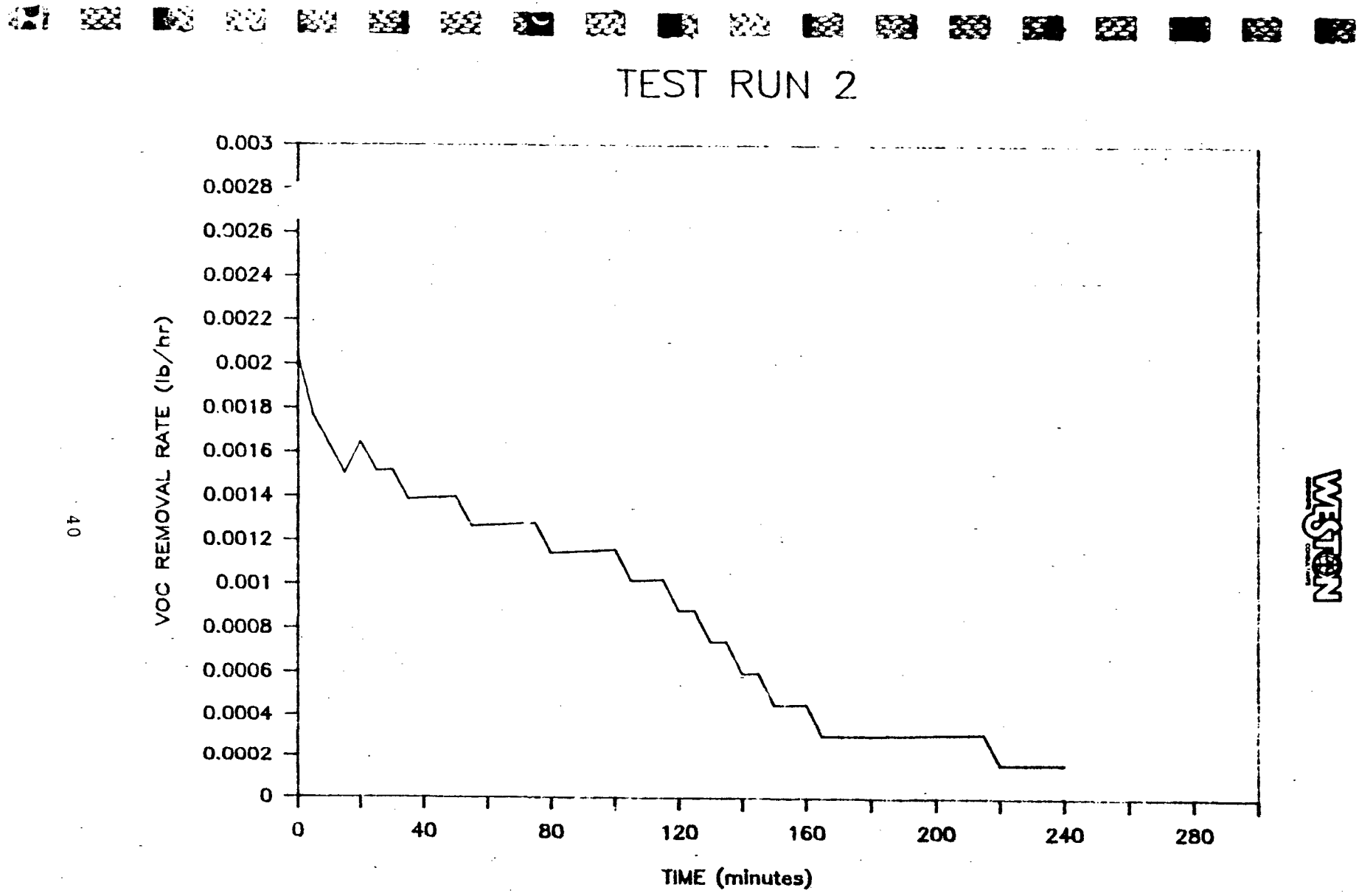

FIGURE 7-4 VOC REMOVAL RATE IN THE DISCHARGE AIR STREAM - TEST RUN 2

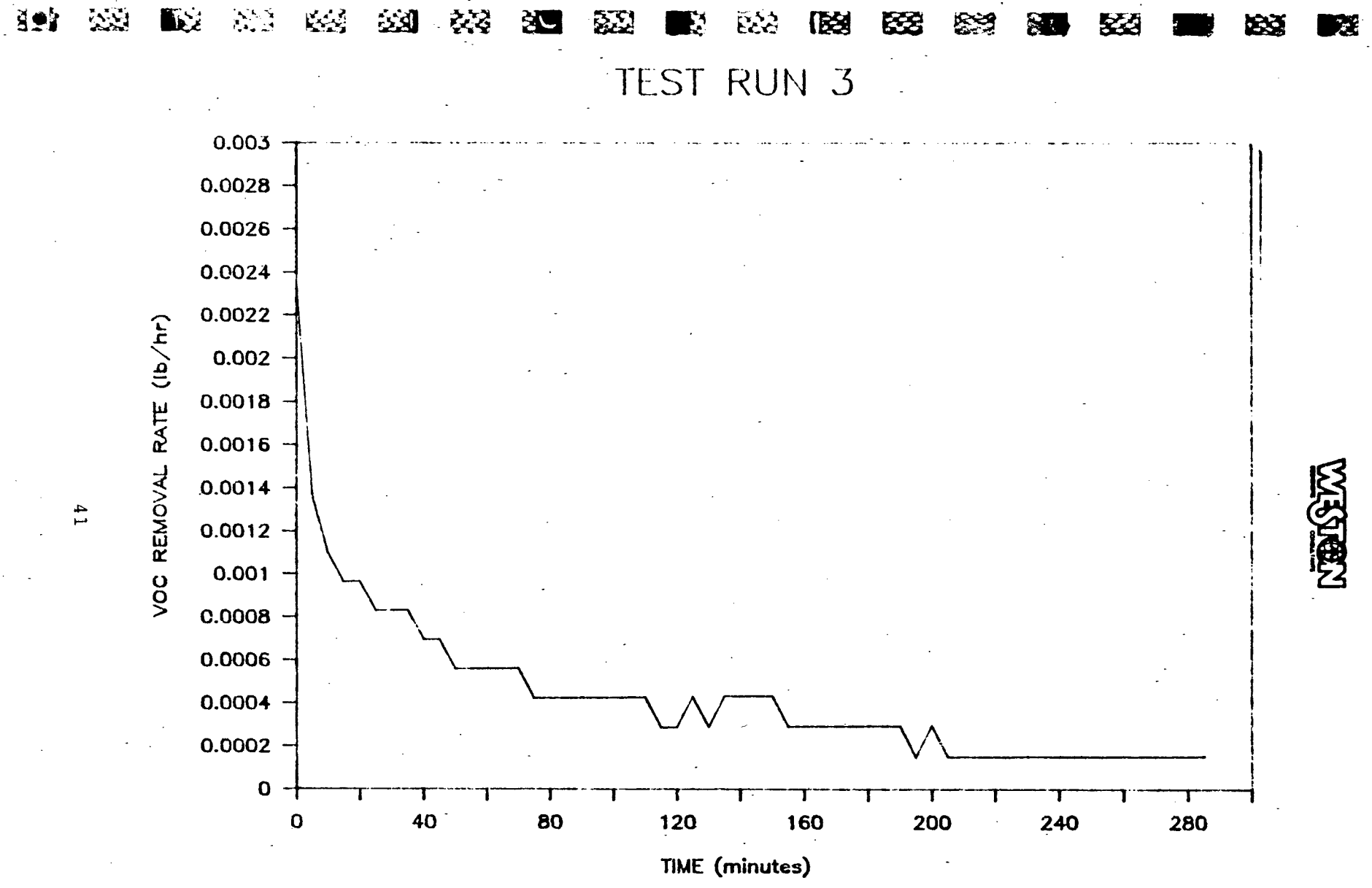

FIGURE 7-5 VOC REMOVAL RATE IN THE DISCHARGE AIR STREAM - TEST RUN 3

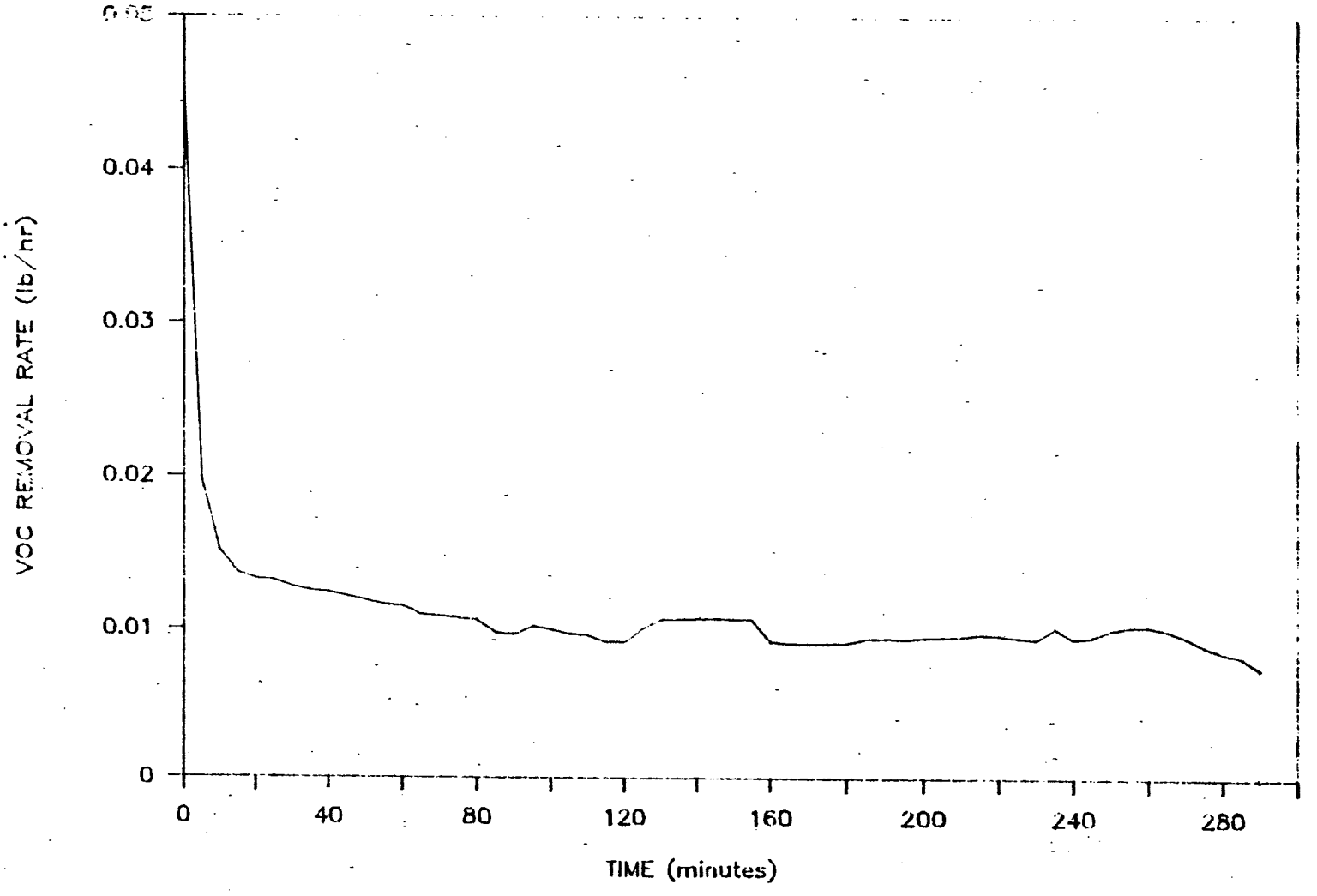

FIGURE 7-6 VOC REMOVAL RATE IN THE DISCHARGE AIR STREAM - TEST RUN 4

8. ANALYSIS OF RESULTS

Analytical results were reviewed to determine the experimental variables that significantly affected VOC removal efficiency. Summaries of pertinent data are contained in Tables 8-1 and 8-2.

Analytical results indicated that VOC removal efficiency is directly related to the total VOC concentration in the feed soils, as shown in Table 8-1. As the feed concentration in each consecutive test run increased, there was a corresponding increase in total VOC removal efficiency. This result is predictable since the driving force for mass transfer is the difference between the VOC concentration in the air stream and the VOC concentration in the soil. Therefore, an increase in the driving force results in an increase in mass transfer and a corresponding increase in VOC removal efficiency. It appears that, for the duration of test periods evaluated (i.e., 230 to 285 minutes), aeration is not sufficient for volatization when the driving force is low (i.e., low VOC concentrations). No conclusion can be made regarding the affect of aeration during much longer test runs (i.e., multiple hours), since extended length runs were not evaluated.

Two operating temperatures were reviewed to determine the effect on VOC removal: 1) the average soil bed temperature and 2) the average inlet air temperature. As shown in Table 8-1 there is no apparent correlation between the soil bed temperature and the VOC removal efficiency. However, there does appear to be a relationship between the inlet air temperature and the VOC removal efficiency. As the inlet air temperature decreased there was a resulting increase in removal efficiency. This correlation suggests that, in this application and with this type of equipment, a lower inlet air temperature improved stripping. However, it may be that the increase in removal efficiency is merely due to the corresponding increase in feed concentration, as discussed above.

The moisture content of the inlet air stream was also evaluated. As shown in Table 8-1, a decrease in the moisture content of the inlet air resulted in an apparent increase in removal efficiency. The explanation for this may be twofold: 1) the drier air had a greater capacity to absorb moisture from the soil; and 2) as the moisture evaporated from the soil the VOC's also evaporated (the VOC's may be in solution in the moisture). This seems to suggest that air with a lower moisture content is more efficient at removing VOC's. However, the correlation is not strong. It may be adviseable to test a broader range of moisture content to further evaluate this effect.

TABLE 8-1 SUMMARY OF OPERATING DATA

Test Run Number	Total VOC Feed Concentration (ug/kg)	Average Soil Bed Temperature (°F)	Average Inlet Air Temperature (°F)	Average Inlet Air Moisture Content (percent by volume)	VOC Removal Efficiency
1	647	105	163	1.90	55
2	1,538	90	144	2.20	70
3	291,940	115	148	0.80	81
4	2,256,100	102	137	1.00	93

Table 8-2 contains the VOC concentrations and soil moisture contents corresponding to Test Runs 3 and 4. VOC removal efficiencies are also included. Analysis of this data indicates that moisture content in the soil is a major indication of VOC removal efficiency. Note that for each test run, the greatest VOC removal occurs when the moisture evaporates from the soil. For Test Run 3, 97.5 percent of the total removal occurred between the time the test started (when the moisture content was 17.6 percent) and at 136 minutes into the test run (when the moisture content was <0.10 percent). A similar trend was followed during test run 4; 96.8 percent of total VOC removal occurred in the first 136 minutes of the run (moisture dropped from 18.8 percent to 3.2 percent). This relationship between moisture content and removal efficiency supports the theory that the majority of VOC's are removed when the moisture evaporates.

TABLE 8-2. SUMMARY OF MOISTURE CONTENT AND REMOVAL EFFICIENCY AS A FUNCTION OF TIME (TEST RUNS 3 AND 4)

	Feed Soil Sample (t = 0)	Intermittent Soil Sample (t = 68 minutes)	Intermittent Soil Sample (t = 136 minutes)	Intermittent Soil Sample (t = 204 minutes)	Treated Soil Sample (t = 285 minutes)
Total VOC Concentration (ug/kg)					
Test Run 3:	291,940	>357,892	60,362	55,365	56,938
Cumulative Removal Efficiency (percent)	---	-23	79	81	81
Moisture Content (percent by weight)	17.6	11.5	<0.10	<0.10	0.5
Total VOC Concentration (ug/kg)					
Test Run 4:	2,256,100	1,936,750	220,640	93,610	158,036
Cumulative Removal Efficiency (percent)	---	14	90	96	93
Moisture Content (percent by weight)	18.8	12.6	3.2	4.4	0.7

--- Not Applicable

9. CONCLUSIONS AND RECOMMENDATIONS

9.1 Conclusions. Based on review of the data associated with all test runs, the following conclusions are presented:

1. Total VOC concentration is directly related to VOC removal efficiency.

2. There is no apparent correlation between the soil bed temperature and VOC removal efficiency.

3. Inlet air temperature appears to be inversely related to VOC removal efficiency.

4. There is no apparent correlation between the moisture content in the inlet air and the VOC removal efficiency.

5. The greatest VOC removal occurs during evaporation of moisture from the soil.

6. Processed soil moisture content provides an indication of VOC removal efficiency and possibly processed soil VOC residuals.

7. Comparison of the VOC removal efficiencies associated with the aeration element and the thermal element (discussed in a separate report[1]) indicates that the role of aeration in thermal stripping is minimal. This conclusion applies to those conditions evaluated in this study (i.e., inlet air pressure, inlet air temperature, inlet air moisture content, ambient air temperature and test duration).

9.2 Recommendations. Based on the results of this field demonstration program, the following recommendations are presented:

1. Apply the conclusions of this report to the evaluation and/or optimization of the thermal stripping process, specifically:

   (a) Utilize a minimal air flow rate since the role of aeration in thermal stripping appears to be minimal.

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[1] Task 11. Pilot Investigation of Low Temperature Thermal Stripping of Volatile Organic Compounds (VOC's) From Soil, Report No. AMXTH-TE-CR-86074, June 1986.

(b) Further evaluate the effects of moisture content in the inlet air stream. Although this study indicated that there is no apparent correlation between the moisture content in the inlet air and the VOC removal efficiency, a very narrow range was evaluated (i.e., 0.8 to 2.2 percent by volume). In future studies, evaluate a broad range of moisture contents (i.e., dehumidified air to saturated air).

(c) Evaluate addition of moisture to soil (i.e., before and during tests to determine the effect on VOC removal efficiency.

(d) Evaluate use of an inert carrier gas (i.e., nitrogen or combustion gases from oil heating unit) instead of air. Although the use of an inert carrier gas is not expected to improve VOC removal efficiency, it will improve the safety of the system (i.e., by avoiding the explosive limits associated with volatile hydrocarbons in air).

2. Evaluate results from Task Order 4, an ongoing benchscale study to investigate in situ volatilization of VOC's from soil, to confirm the findings of this study.

3. Conduct bench/pilot studies to further evaluate the effect of operating parameters on VOC removal efficiency (i.e., a greater range of temperatures, different soil bed heights, a variety of moisture contents in air, etc.).

4. Further investigate the correlation between processed soil moisture content and VOC concentration to determine if soil moisture content could be used to monitor, predict, and/or control soil VOC decontamination effectiveness. During investigations, the soil moisture content and VOC concentration should be monitored before, during, and after aeration to determine if a correlation exists.

WESTON

APPENDICES

APPENDIX A - ORGANIC WASTE CHARACTERISTICS OF SITE SOILS AT LEAD (DETERMINED DURING PRELIMINARY INVESTIGATIONS)

APPENDIX B - GRAIN SIZE GRADATION CURVES CORRESPONDING TO FILL SOIL AND NATIVE SOIL

APPENDIX C - ANALYTICAL METHODS

APPENDIX D - SUPPLEMENTAL DATA

APPENDIX A

ORGANIC WASTE CHARACTERISTICS OF SITE SOILS AT LEAD
(DETERMINED DURING PRELIMINARY INVESTIGATIONS)

TABLE A-1. VOLATILE ORGANIC COMPOUNDS (VOC'S) INCLUDED ON THE HAZARDOUS SUBSTANCE LIST (HSL)

Volatile organic compounds	Detection limits* Low water[a] ug/L	Low soil/ sediment[b] ug/Kg
1. Chloromethane	10	10
2. Bromomethane	10	10
3. Vinyl Chloride	10	10
4. Chloroethane	10	10
5. Methylene Chloride	5	5
6. Acetone	10	10
7. Carbon Disulfide	5	5
8. 1,1-Dichloroethene	5	5
9. 1,1-Dichloroethane	5	5
10. Trans-1,2-Dichloroethene	5	5
11. Chloroform	5	5
12. 1,2-Dichloroethane	5	5
13. 2-Butanone	10	10
14. 1,1,1-Trichloroethane	5	5
15. Carbon Tetrachloride	5	5
16. Vinyl Acetate	10	10
17. Bromodichloromethane	5	5
18. 1,1,2,2-Tetrachloroethane	5	5
19. 1,2-Dichloropropane	5	5
20. Trans-1,3-Dichloropropene	5	5
21. Trichloroethene	5	5
22. Dibromochloromethane	5	5
23. 1,1,2-Trichloroethane	5	5
24. Benzene	5	5
25. Cis-1,3-Dichloropropene	5	5

[a] Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Water CRDL.

[b] Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Soil/Sediment CRDL.

*Detection limits listed for soil/sediment are based on wet weight.

TABLE A-1. (CONTINUED)

Volatile organic compounds	Detection limits* Low water[a] ug/L	Low soil/ sediment[b] ug/Kg
26. 2-Chloroethyl Vinyl Ether	10	10
27. Bromoform	5	5
28. 2-Hexanone	10	10
29. 4-Methyl-2-pentanone	10	10
30. Tetrachloroethene	5	5
31. Toluene	5	5
32. Chlorobenzene	5	5
33. Ethyl Benzene	5	5
34. Styrene	5	5
35. Total Xylenes	5	5

[a]Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Water CRDL.

[b]Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Soil/Sediment CRDL.

*Detection limits listed for soil/sediment are based on wet weight.

TABLE A-2. CONCENTRATION RANGE OF VOLATILE ORGANIC COMPOUNDS (VOC'S) DETERMINED TO BE PRESENT IN AREA K-1 (BASED ON TESTING PERFORMED ON 10-12 JUNE 1985)*

Compound	Concentration (ug/g)** Borehole 1	Borehole 2	Borehole 3	Borehole 4
1. Volatiles on Hazardous Substance List (HSL)				
Acetone				
Benzene				
Bromomethane				
Bromoform				
2-Butanone				
Carbon Disulfide				
Carbon Tetrachloride				
Chlorobenzene	0.33-240			
Chlorodibromomethane				
Chloroethane				
2-Chloroethylvinyl Ether				
Chloroform				
Chloromethane				
Dichlorobromomethane				
1,1-Dichloroethane				
1,2-Dichloroethane				
1,1-Dichloroethylene				
1,2-Dichloropropane				
1,3-Trans Dichloropropene				
1,3-Cis Dichloropropene				
Ethylbenzene	3.5-4.8	0-3.7	0.73-5.9	0-0.002
2-Hexanone				
Methylene Chloride	0-4.3			
4-Methyl-2-Pentanone				
Styrene				
1,1,2,2-Tetrachloroethane				
Tetrachloroethylene	0.39-28	0.012-0.6	0.008-29	0-0.047
Toluene	0-16			0-0.002
1,2-cis/trans Dichloroethylene	5.8->1300	0.03-76	13-390	0.07-4.8

*For reference, the locations of soil borings drilled in Area K-1 during the waste characterization phase of the pilot study are shown in Figure A-1.

**Concentration ranges correspond to the minimum and maximum concentrations observed for all discrete samples (i.e., 1.5'-3.5', 3.5'-5.0', 5.0'-6.5', 6.5'-8.0', 8.0'-10.0').

TABLE A-2. (CONTINUED)

Compound	Concentration (ug/g)** Borehole 1	Borehole 2	Borehole 3	Borehole 4
1. Volatiles on Hazardous Substance List (HSL) (continued)				
1,1,2-Trichloroethane				
1,1,1-Trichloroethane			0-14	
Trichloroethylene	0.84-16	0.03-27	0.078-300	0.02-1.1
Vinyl Acetate				
Vinyl Chloride	0-2.1		0-2.6	
Xylene	25-32	0.006-25	4-31	0-0.006
Total Volatiles	35.86-1643.2	0.078-132.3	17.816-772.5	0.09-5.957
2. Others				
Cio-Allyl Benzene	20-30			
Dichlorobenzene	3-600	0.03-10	0.009-100	0-0.07
Methyl Ethyl Benzene	0.07-30	0-10	2.3-9	
n-Propylbenzene	4-7	0-3	0-2.9	
Trimethyl Benzene	30-110	0.13-60	8.4-37	
Total Others	57.07-777	0.16-83	10.709-148.9	0-0.07
TOTAL	92.93-2420.2	0.238-215.3	28.525-921.4	0.09-6.027

*For reference, the locations of soil borings drilled in Area K-1 during the waste characterization phase of the pilot study are shown in Figure A-1.

**Concentration ranges correspond to the minimum and maximum concentrations observed for all discrete samples (i.e., 1.5'-3.5', 3.5'-5.0', 5.0'-6.5', 6.5'-8.0', 8.0'-10.0').

TABLE A-2. (CONTINUED)

Compound	Concentration (ug/g)** Borehole 5	Borehole 6	Borehole 7	Borehole 8
1. Volatiles on Hazardous Substance List (HSL)				
Acetone				
Benzene		0-0.28		
Bromomethane				
Bromoform				
2-Butanone				
Carbon Disulfide				
Carbon Tetrachloride				
Chlorobenzene		0-0.44		
Chlorodibromomethane				
Chloroethane				
2-Chloroethylvinyl Ether				
Chloroform				
Chloromethane				
Dichlorobromomethane				
1,1-Dichloroethane		0-0.26		
1,2-Dichloroethane				
1,1-Dichloroethylene		0.3-2.7		0-1.8
1,2-Dichloropropane				
1,3-Trans Dichloropropene				
1,3-Cis Dichloropropene				
Ethylbenzene		0.97-4.3	0-4.9	0.15-11
2-Hexanone				
Methylene Chloride	0-1.7	0-0.6		
4-Methyl-2-Pentanone				
Styrene				
1,1,2,2-Tetrachloroethane		0.07-0.76		
Tetrachloroethylene	0.012-0.064	0.009-4.2	210->3800	0.058-17
Toluene		4.9-8.2		
1,2-cis/trans Dichloroethylene	0.46-5.2	0.098-990	10-130	0.9-920

*For reference, the locations of soil borings drilled in Area K-1 during the waste characterization phase of the pilot study are shown in Figure A-1.

**Concentration ranges correspond to the minimum and maximum concentrations observed for all discrete samples (i.e., 1.5'-3.5', 3.5'-5.0', 5.0'-6.5', 6.5'-8.0', 8.0'-10.0').

TABLE A-2. (CONTINUED)

Compound	Concentration (ug/g)** Borehole 5	Borehole 6	Borehole 7	Borehole 8
1. Volatiles on Hazardous Substance List (HSL) (continued)				
1,1,2-Trichloroethane				
1,1,1-Trichloroethane			34-48	
Trichloroethylene	0.047-1.2	0.056-330	25->3500	1.2-3000
Vinyl Acetate				
Vinyl Chloride		0-4.3		4.4-4.8
Xylene		0.049-25	5.1-24	0.82-47
Total Volatiles	0.519-8.164	6.452 1371.04	284.1- 7506.9	7.528 4001.6
2. Others				
Cio-Allyl Benzene		2-20		0-5
Dichlorobenzene	0-0.4	7-200	0.9-2.4	0.5-20
Methyl Ethyl Benzene		0.5-24	0-10	0.4-11
n-Propylbenzene		0.72-5.6		0-4
Trimethyl Benzene		3.7-66	0-43	2.5-50
Total Others	0-0.4	13.92- 315.6	0.9-55.4	3.4-90
TOTAL	0.519-8.564	20.372 1686.64	285- 7562.3	10.928 4091.6

*For reference, the locations of soil borings drilled in Area K-1 during the waste characterization phase of the pilot study are shown in Figure A-1.

**Concentration ranges correspond to the minimum and maximum concentrations observed for all discrete samples (i.e., 1.5'-3.5', 3.5'-5.0', 5.0'-6.5', 6.5'-8.0', 8.0'-10.0').

TABLE A-2. (CONTINUED)

Compound	Concentration (ug/g)** Borehole 9	Borehole 10	Borehole 11
1. Volatiles on Hazardous Substance List (HSL)			
Acetone			
Benzene			
Bromomethane			
Bromoform			
2-Butanone			
Carbon Disulfide			
Carbon Tetrachloride			
Chlorobenzene			
Chlorodibromethane			
Chloroethane			
2-Chloroethylvinyl Ether			
Chloroform			
Chloromethane		0-0.1	
Dichlorobromomethane			
1,1-Dichloroethane			
1,2-Dichloroethane			
1,1-Dichloroethylene	0-0.01		
1,2-Dichloropropane			
1,3-Trans Dichloropropene			
1,3-Cis Dichloropropene			
Ethylbenzene			
2-Hexanone			
Methylene Chloride			
4-Methyl-2-Pentanone			
Styrene			
1,1,2,2-Tetrachloroethane			
Tetrachloroethylene	0.006-170	0.016-0.83	0-0.007
Toluene			0-0.006
1,2-cis/trans Dichloroethylene	4.5-74	0.05-0.08	0.007-0.023

*For reference, the locations of soil borings drilled in Area K-1 during the waste characterization phase of the pilot study are shown in Figure A-1.

**Concentration ranges correspond to the minimum and maximum concentrations observed for all discrete samples (i.e., 1.5'-3.5', 3.5'-5.0', 5.0'-6.5', 6.5'-8.0', 8.0'-10.0').

TABLE A-2. (CONTINUED)

Compound	Concentration (ug/g)** Borehole 9	Borehole 10	Borehole 11
1. Volatiles on Hazardous Substance List (HSL) (continued)			
1,1,2-Trichloroethane			
1,1,1-Trichloroethane	0-30		
Trichloroethylene	0.14-1700	0.01-2.5	0.012-0.037
Vinyl Acetate			
Vinyl Chloride		0.05-0.24	
Xylene	8-11	0.012-0.06	
Total Volatiles	12.646 1985.01	0.138-3.81	0.019-0.073
2. Others			
Cis-Allyl Benzene		0-0.08	
Dichlorobenzene	2-11	0.02-0.1	
Methyl Ethyl Benzene	0-4	0.02-0.13	
n-Propylbenzene		0-0.02	
Trimethyl Benzene	0-20	0.13-0.44	
Total Others	2-35	0.17-0.77	
TOTAL	14.646 2020.01	0.308-4.58	0.019-0.073

*For reference, the locations of soil borings drilled in Area K-1 during the waste characterization phase of the pilot study are shown in Figure A-1.

**Concentration ranges correspond to the minimum and maximum concentrations observed for all discrete samples (i.e., 1.5'-3.5', 3.5'-5.0', 5.0'-6.5', 6.5'-8.0', 8.0'-10.0').

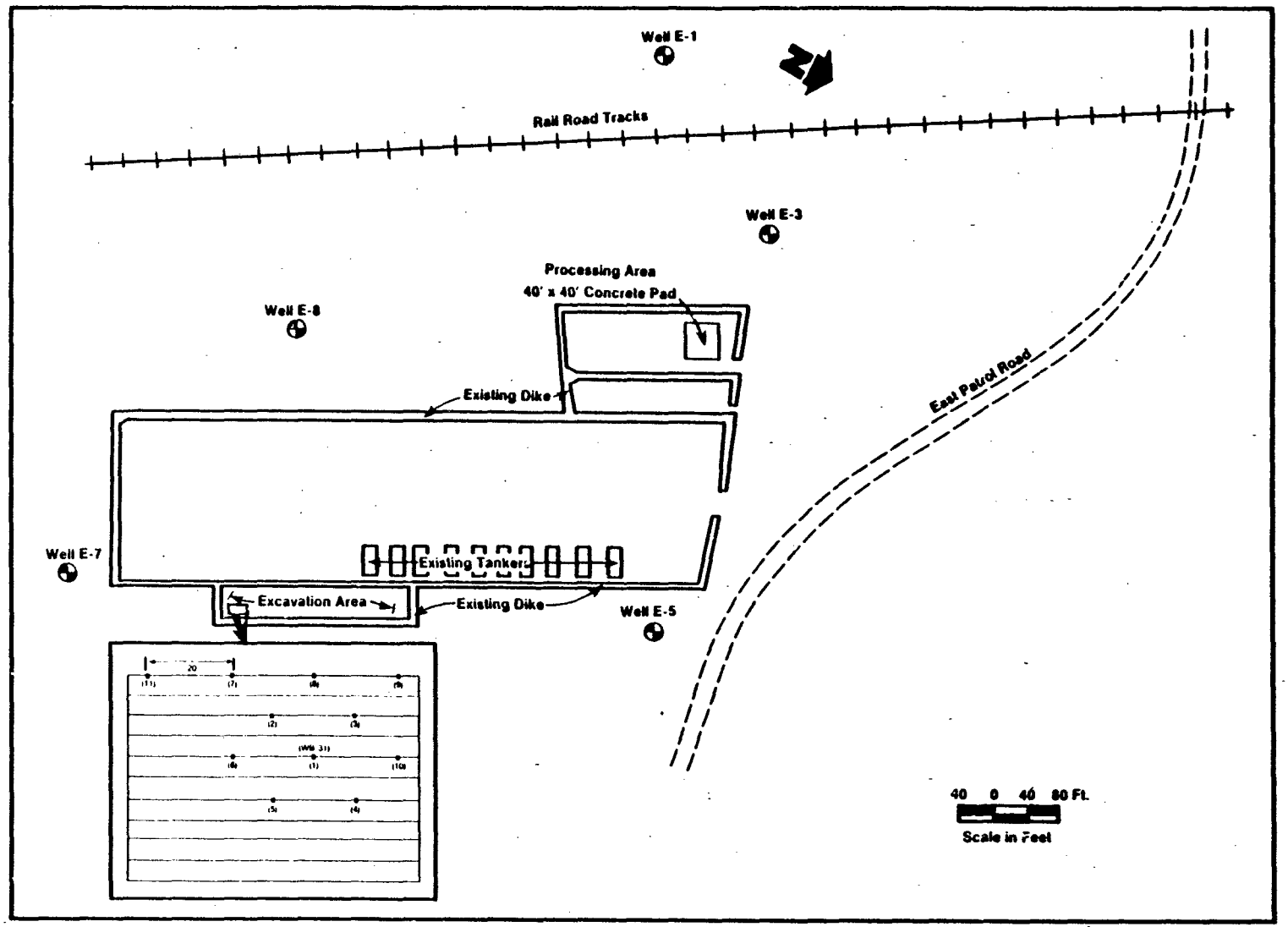

FIGURE A-1 LOCATION OF SOIL BORINGS DRILLED IN AREA K-1 DURING THE WASTE CHARACTERIZATION PHASE OF THE PILOT STUDY

TABLE A-3. VOC CONCENTRATIONS IN EXCAVATED SOILS FROM PHASE 1 OF THE PILOT INVESTIGATION (PPM BY WEIGHT)

Test Run No.	Dichloroethylene	Trichloroethylene	Tetrachloroethylene	Xylene	Other VOC's	Total VOC's
I. Phase I Test Runs						
1	0.48	0.64	0.13*	0.12*	0.03*	1.40
2	110.00	3,600.00	4,800.00	35.00*	10.40*	8,555.40
3	3.10	1.50	4.70	0.26	0.06*	9.62
4	0.21	0.29	0.81	BDL	0.04*	1.35
5	830.00	20,000.00	580.00	460.00	117.00*	21,987.00
6	770.00	8,400.00	39.00*	240.00	56.00*	9,505.00
7	1.20	1.50	0.64*	BDL	0.62*	4.16
8	110.00	1,200.00	190.00	97.00*	12.05*	1,609.05
9	1,200.00	2,640.00	BDL	47.00*	269.60	4,156.60
10	270.00	2,200.00	1,300.00	110.00	26.60*	3,906.60
11	100.00	830.00	530.00	60.00	17.30*	1,537.30
12				NO EXCAVATION		
13	62.00	39.00*	30.00*	29.00*	BDL	160.00
14	130.00	1,600.00	230.00	150.00	28.30*	2,138.30
15	310.00	2,200.00	2,300.00	140.00	35.00*	4,985.00
16	140.00	950.00	1,900.00	13.00*	40.80	3,043.80
17				NO EXCAVATION		
18	BDL	BDL	8.00*	BDL	BDL	8.00
Average	252.30	2,728.90	744.60	86.30	38.40	3,850.60

*Estimated value
BDL = Below Detection Limit

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TABLE A-3. (CONTINUED)

Test Run No.	Dichloroethylene	Trichloroethylene	Tetrachloroethylene	Xylene	Other VOC's	Total VOC's
II. Phase II Test Runs						
19	1.80*	BDL	BDL	6.30	1.50*	9.60
20			NO EXCAVATION			
21	0.02*	0.08*	0.03*	0.10	BDL	0.22
22	0.45*	BDL	BDL	79.00	34.76	114.21
23			NO EXCAVATION			
24	74.00	>390.00	>260.00	>7,190.00	16.80	>930.80
25			NO EXCAVATION			
26			NO EXCAVATION			
27	13.00*	340.00	210.00	35.00*	BDL	598.00
28	———	———	———	———	———	———
Average	17.85	>146.02	>94.01	>62.08	10.61	>330.57

*Estimated Value
BDL - Below Detection Limit

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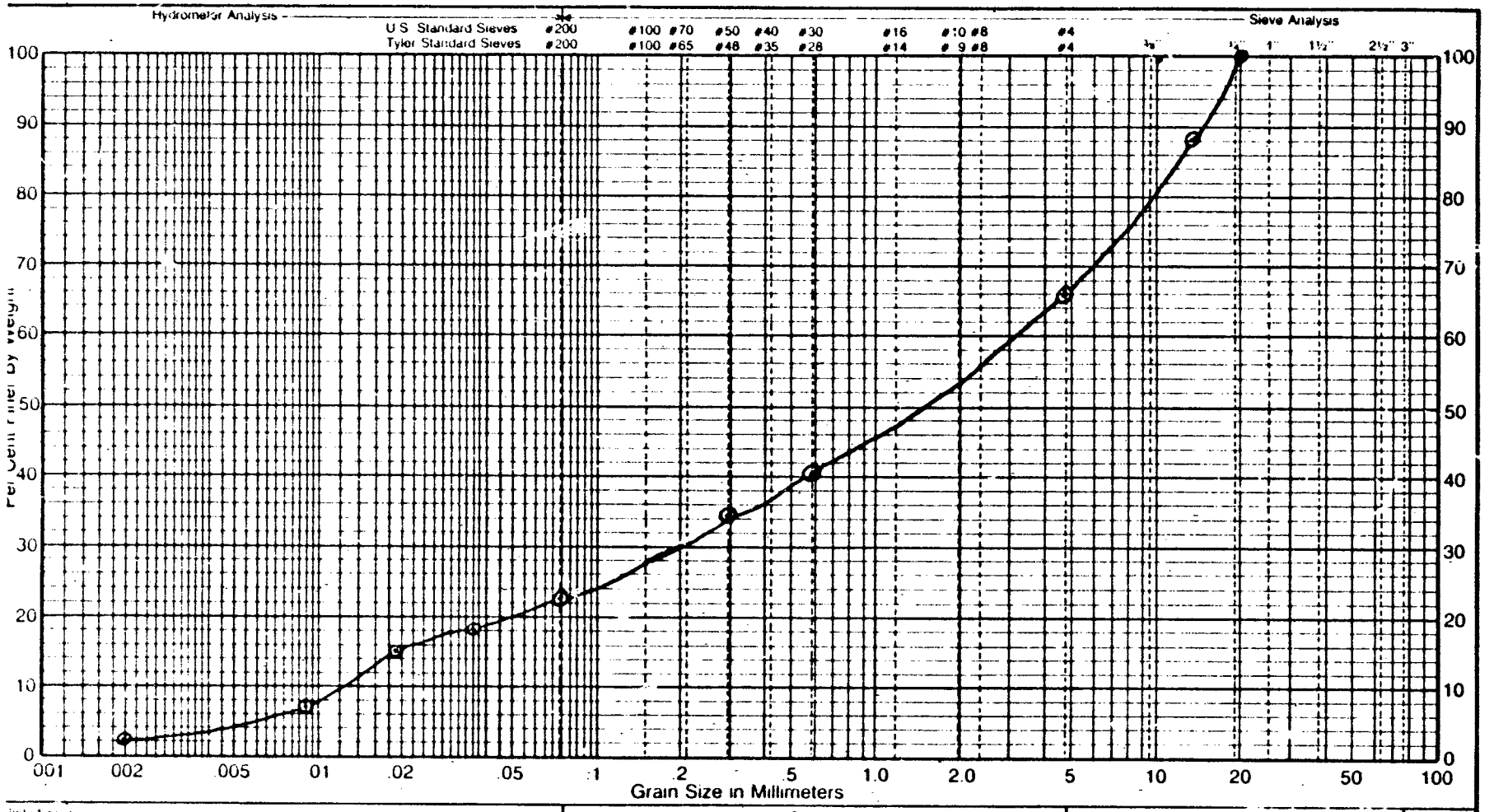
APPENDIX B

GRAIN SIZE GRADATION CURVES CORRESPONDING TO FILL SOIL AND NATIVE SOIL



Symbol	Sample	D60	Specific Gravity	Description of Sample
				FILL - GRAVELLY SAND

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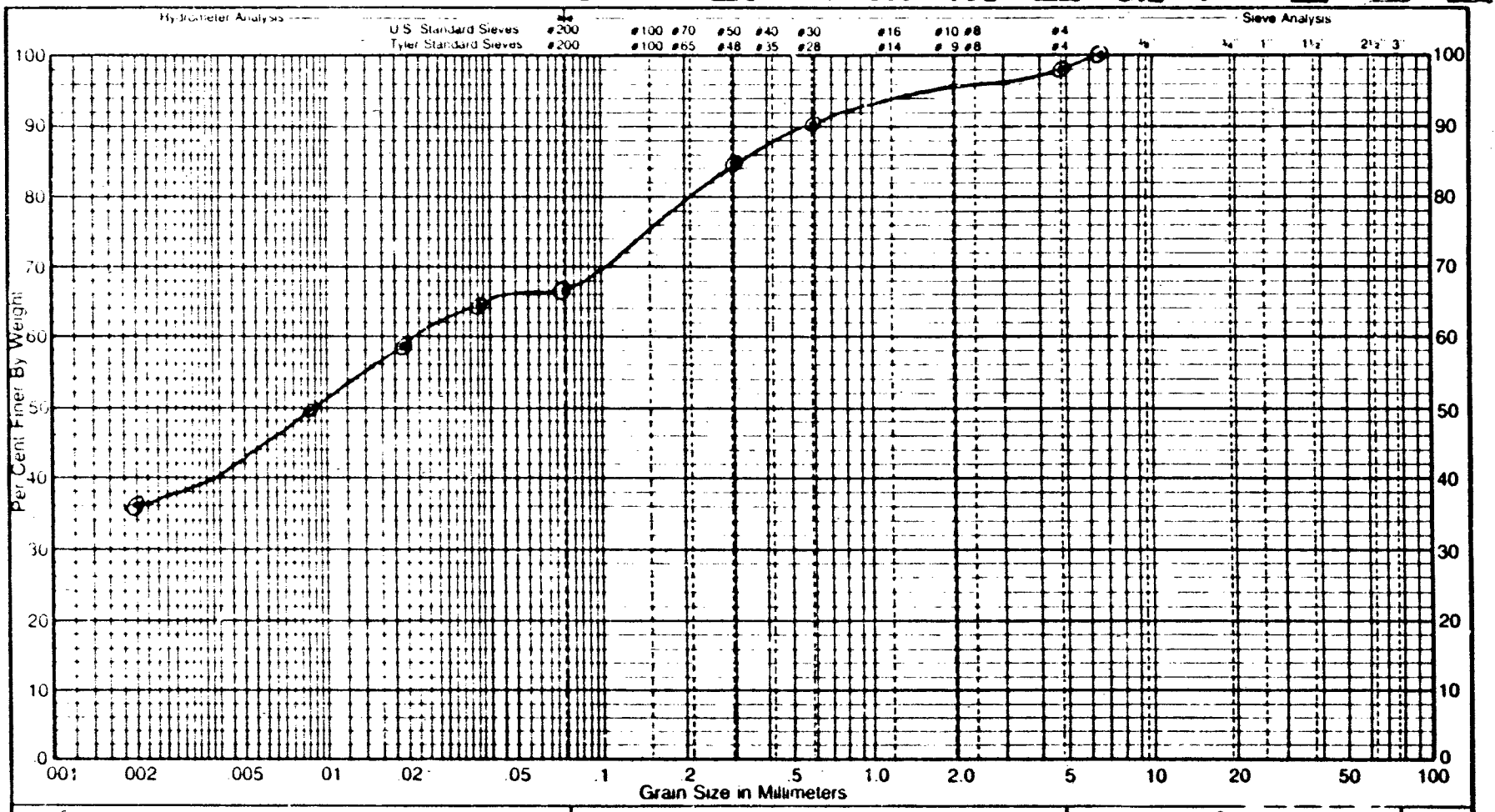
Gradation Curves



Symbol	Sample	D60	Specific Gravity	Description of Sample
B-2				Native Soil - Sandy CLAY/Sandy SILT

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Gradation Curves

APPENDIX C

ANALYTICAL METHODS

- EPA CONTRACT LABORATORY PROTOCOL FOR GC/MS ANALYSIS, PURGEABLE ORGANICS IN WATER, SOILS AND SEDIMENTS

- STANDARD METHOD 209G

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EPA CONTRACT LABORATORY PROTOCOL FOR GC/MS ANALYSIS PURGEABLE ORGANICS IN WATER, SOILS, AND SEDIMENTS

6060A

1. GC/MS Analysis of Purgeable Organics

1.1 Summary of Methods

1.1.1 Water samples

An inert gas is bubbled through a 5 mL sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

An aliquot of the sample is diluted with reagent water when dilution is necessary. A 5 mL aliquot of the dilution is taken for purging.

1.1.2 Sediment/Soil Samples

1.1.2.1 Low Level. An inert gas is bubbled through a mixture of a 5 gm sample and reagent water contained in a suggested specially designed purging chamber (illustrated on page D-95) at elevated temperatures. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is complet[illegible], the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

1.1.2.2 Medium Level. A measured amount of soil is extracted with methanol. A portion of the methanol extract is diluted to 5 mL with reagent water. An inert gas is bubbled through this solution in a specifically designed purging chamber at ambient temperature. The purgeables are effectively transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purbeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.

1.2 Interferences

1.2.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Exhibit E. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

1.2.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling. A holding blank prepared from reagent water and carried through the holding period and the analysis protocol serves as a check on such contamination. One holding blank per case must be analyzed.

1.2.3 Contamination by carry over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry over, the purging device and sampling syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

1.3 Apparatus and Materials

1.3.1 Micro syringes - 25 uL and larger, 0.006 inch ID needle.

1.3.2 Syringe valve - two-way, with Luer ends (three each), if applicable to the purging device.

1.3.3 Syringe - 5 mL, gas tight with shut-off valve.

1.3.4 Balance-Analytical, capable of accurately weighing 0.0001 g. and a top-loading balance capable of weighing 0.1g.

1.3.5 Glassware

1.3.5.1
- o Bottle - 15 mL, screw cap, with Teflon cap liner.
- o Volumetric flasks - class A with ground-glass stoppers.
- o Vials - 2 mL for GC autosampler.

1.3.6 Purge and trap device - The purge and trap device consists of three separate pieces of equipment; the sample purger, trap and the desorber. Several complete devices are now commercially available.

1.3.6.1 The sample purger must be designed to accept 5 mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The sample purger, illustrated in Figure 1, meets these design criteria. Alternate sample purge devices may be utilized provided equivalent performance is demonstrated.

1.3.6.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following minimum lengths of absorbents: 1.0 cm of methyl silicone coated packing (3% OV-1 on Chromosorb W or equivalent), 15 cm of 2,6-diphenylene oxide polymer (Tenax-GC 60/80 mesh) and 8 cm of silica gel (Davison Chemical, 35/60 mesh, grade 15, or equivalent). The minimum specifications for the trap are illustrated in Figure 2.

1.3.6.3 The desorber should be capable of rapidly heating the trap to 180°C. The polymer section of the trap should not be heated higher than 180°C and the remaining sections should not exceed 220°C. The desorber design, illustrated in Figure 2, meets these criteria.

1.3.6.4 The purge and trap device may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

1.3.6.5 A heater or heated bath capable of maintaining the purge device at 40°C ± 1°C.

1.3.7 GC/MS system

1.3.7.1 Gas chromatograph - An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

1.3.7.2 Column - 6 ft long x 0.1 in ID glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent.

1.3.7.3 Mass spectrometer - Capable of scanning from 35 to 260 amu every seven seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum which meets all the criteria in table 2 when 50 ng of 4-bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

1.3.7.4 GC/MS interface - Any gas chromatograph to mass spectrometer interface that gives acceptable calibration points at 50 ng or less per injection for each of the parameters of interest and achieves all acceptable performance criteria (Exhibit E) may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

1.3.7.5 Data system - A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any ECIP between specified time or scan number limits.

1.4 Reagents

1.4.1 Reagent water - Regent water is defined as water in which an interferent is not observed at the MDL of the parameters of interest.

1.4.1.1 Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

1.4.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

1.4.1.3 Reagent water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90°C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a narrow-mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

1.4.2 Sodium thiosulfate - (ACS) Granular.

1.4.3 Methanol - Pesticide quality or eqvuivalent.

1.4.4 Stock standard solutions - Stock standard solutions may be prepared from pure standard materials or purchased and must be traceable to EMLS/LV supplied standards. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate.

1.4.4.1 Place about 9.8 mL of methanol into a 10.0 mL tared ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

1.4.4.2 Add the assayed reference material as described below.

1.4.4.2.1 Liquids - Using a 100 uL syringe, immediately add two or more drops of assayed reference material to the flask then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

1.4.4.2.2 Gases - To prepare standards for any of the four halocarbons that boil below 30°C (bromomethane, chloroethane, chloromethane, and vinyl chloride), fill a 5 mL valved gas-tight syringe with the reference standard to the 5.0 mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid. The heavy gas rapidly dissolves in the methanol.

1.4.4.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standards may be used at any concentration if they are certified by the manufacturer. Commercial standards must be traceable to EMSL/LV supplied standards.

1.4.4.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace at -10°C to -20°C and protect from light.

1.4.4.5 Prepare fresh standards weekly for the four gases and 2-chloroethyl-vinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicate a problem.

1.4.5 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. (See GC/MS Calibration in Exhibit E). Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

1.4.6 Surrogate standard spiking solution. Prepare stock standard solutions for toluene-d8, p-bromofluorobenzene, and 1,2-dichloroethane-d4 in methanol as described in Paragraph 1.4.4. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 250 ug/10 mL in methanol.

1.4.7 Purgeable Organic Matrix Standard Spiking Solution

1.4.7.1 Prepare a spiking solution in methanol that contains the following compounds at a concentration of 250 ug/10.0 mL:

Purgeable Organics

1,1-dichloroethene
trichloroethene
chlorobenzene
toluene
benzene

1.4.7.2 Matrix spikes also serve as duplicates; therfore, add an aliquot of this solution to each of two portions from one sample chosen for spiking.

1.4.8 BFB Standard - Prepare a 25 ng/uL solution of BFB in methanol.

1.4.9 Great care must be taken to maintain the integrity of all standard solutions. It is recommended that all standard solutions be stored at -10°C to -20°C in screw cap amber bottles with teflon liners.

1.5 Calibration

1.5.1 Assemble a purge and trap device that meets the specification in paragraph 1.3.6. Condition the trap overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, daily condition traps 10 minutes while backflushing at 180°C with the column at 220°C.

1.5.2 Connect the purge and trap device to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate parameters equivalent to those in paragraph 1.7.1.2 Calibrate the purge and trap-GC/MS system using the internal standard technique (paragraph 1.5.3).

1.5.3 Internal standard calibration procedure. The three internal standards are bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5.

1.5.3.1 Prepare calibration standards at a minimum of five concentration levels for each HSL parameter. The concentration levels are specified in Exhibit E. Aqueous standards may be stored up to 24 hours, if held in sealed vials with zero headspace as described in paragraph 1.7. If not so stored, they must be discarded after an hour.

1. .3.2 Prepare a spiking solution containing each of the internal standards using the procedures described in paragraphs 1.4.4 and 1.4.5. It is recommended that the secondary dilution standard be prepared at a concentration of 25 ug/mL of each internal standard compound. The addition of 10 uL of this standard to 5.0 mL of sample or calibration standard would be equivalent of 50 ug/L.

1.5.3.3 Analyze each calibration standard, according to paragraph 1.7 adding 10 uL of internal standard spiking solution directly to the syringe. Tabulate the area response of the characteristic ions against concentration for each compound and internal standard and calculate response factors (RF) for each compound using equation 1.

EQ. 1 $$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard from Exhibit E.

C_{is} = Concentration of the internal standard.

C_x = Concentration of the compound to be measured.

1.5.3.4 The average response factor (RF) must be calculated for all compounds. A system performance check must be made before this calibration curve is used. Five compounds (the system performance check compounds) are checked for a minimum average response factor. These compounds (the SPCC) are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. Five compounds (the calibration check compounds, CCC) are used to evaluate the curve. Calculate the % Relative Standard Deviation (%RSD) of RF values over the working range of the curve. A minimum %RSD for each CCC must be met before the curve is valid.

$$\%RSD = \frac{\text{Standard deviation}}{\text{mean}} \times 100$$

See instructions for Form VI, Initial Calibration Data for more details.

1.5.3.5 Check of the calibration curve should be performed once every 12 hours. These criteria are described in detail in the instructions for Form VII, Continuing Calibration Check. The minimum response factor for the system performance check compounds must be checked. If this criteria is met, the response factor of all

compounds a.e calculated and reported. A percent difference of the daily response factor (12 hour) compared to the average response factor from the initial curve is calculated. The maximum percent difference allowed for each compound flagged as 'CCC' in Form VII is checked. Only after both these criteria are met can sample analysis begin.

1.5.3.6 Internal standard responses and retention times in all samples must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions and corrections made as required. If the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is necessary. Retention time and EICP area records shall be maintained in appropriate form by the laboratory as a part of its internal quality control (Exhibit E).

1.6 GC/MS Operating Conditions

1.6.1 These performance tests require the following instrumental parameters:

Electron Energy:	70 Volts (nominal)
Mass Range:	35 - 260
Scan Time:	to give at least 5 scans per peak but not to exceed 7 seconds per scan.

→ Will be changed to Something like 10 scans/peak 5/84 2 seconds/scan.

1.7 Sample Analysis

1.7.1 Water Samples

1.7.1.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

1.7.1.2 Recommended operating conditions for the gas chromatograph - Column conditions: Carbopak B (60/80 mesh with 1% SP-1000 pakced in a 6 foot by 2 mm ID glass column with helium carrier gas at a flow rate of 30 mL/min. Column temperature is isothermal at 45°C for 3 minutes, then programmed at 8°C per minute to 220°C and held for 15 minutes.

1.7.1.3 After achieving the key ion abundance criteria, calibrate the system daily as described in Exhibit E.

1.7.1.4 Adjust the purge gas (helium) flow rate to 40 ± 3 mL/min. Variations from this flow rate may be necessary to achieve better purging and collection efficiencies for some compounds, particularly chloromethane and bromoform.

1.7.1.5 Remove the plunger from a 5 mL syringe and attach a closed syringe valve. Open the sample or standard bottle which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the sample for future analysis so if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such a time when the

analyst has determined that the first sample has been analyzed properly. Filling one 20 mL syringe would allow the use of only one syringe. If a second analysis is needed from the 20 mL syringe, it must be analyzed within 24 hours. Care must also be taken to prevent air from leaking into the syringe.

1.7.1.6 The purgeable organics screening procedure (Section III, paragraph 1.0), if used, will have shown the approximate concentrations of major sample components. If a dilution of the sample was indicated, this dilution shall be made just prior to GC/MS analysis of the sample.

1.7.1.6.1 The following procedure will allow for dilutions near the calculated dilution factor from the screening procedure:

- All dilutions are made in volumetric flasks (10 mL to 100 mL).
- Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.
- Calculate the approximate volume of reagent water which will be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.
- Inject the proper aliquot from the syringe prepared in paragraph 1.7.1.5 into the volumetric flask. Aliquots of less than 1 mL increments are prohibited. Dilute the flask to the mark with reagent water. Cap the flask, invert, and shake three times.
- Fill a 5 mL syringe with the diluted sample as in paragraph 1.7.1.5.

- If this is an intermediate dilution, use it and repeat above procedure to achieve larger dilutions.

1.7.1.7 Add 10.0 uL of the surrogate spiking solution (1.4.6) and 10.0 uL of the internal standard spiking solution (1.5.3.2) through the valve bore of the syringe, then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10 uL of the surrogate spiking solution to 5mL of sample is equivalent to a concentration of 50 ug/L of each surrogate standard.

1.7.1.8 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

1.7.1.9 Close both valves and purge the sample for 12.0 $\pm$ 0.1 minutes at ambient temperature.

1.7.1.10 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 180°C while backflushing the trap with an inert gas between 20 and 60 mL/min for four minutes. If this rapid heating requirement cannot be met, the gas chromatographic column must be used as a secondary trap by cooling it to 30°C (or subambient, if problems persist) instead of the recommended initial temperature of 45°C.

1.7.1.11 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 mL flushes of reagent water to avoid carry-over of pollutant compounds.

1.7.1.12 After desorbing the sample for four minutes, recondition the trap by returning the purge and trap device to the purge mode. Wait 15 seconds then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180°C. Trap temperatures up to 230°C may be employed, however the higher temperature will shorten the useful life of the trap. After approximately seven minutes turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

1.7.1.13 If the initial analysis of a sample or a dilution of a sample indicates saturated ions of HSL compounds, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

1.7.1.14 For low and medium level water samples, add 10 uL of the matrix spike solution (1.4.7) to the 5mL of sample purged. Disregarding any dilutions, this is equivalent to a concentration of 50 ug/L of each matrix spike standard.

1.7.1.15 All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

1.7.2 Sediment/Soil Samples

Two approaches may be taken to determine whether the low level or medium level method may be followed.

- o Assume the sample is low level and analyze a 5 gram sample
- o Use the X factor calculated from the optional Hexadecane screen (Section III), paragraph 1.7.2.1.3

If any peaks are saturated from the analysis of a 5 gram sample, a smaller sample size must be analyzed to prevent saturation. However, the smallest sample size permitted is 1 gm. If smaller than 1 gram sample size is needed to prevent saturation, the medium level method must be used.

1.7.2.1 Low Level Method

The low level method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and internal standards.

Use 5 grams of sample or use the X Factor to determine the sample size for purging.

- o If the X Factor is 0 (no peaks noted on the hexadecane screen), analyze a 5 gm sample.
- o If the X Factor is between 0 and 1.0, analyze a 1 gm sample.

1.7.2.1.1 The GC/MS system should be set up as in 1.7.1.2 - 1.7.1.4. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and sample.

1.7.2.1.2 Remove the plunger from a 5 mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 uL each of the surrogate spiking solution (1.4.6) and the internal standard solution to the syringe through the valve. (Surrogate spiking solution and internal standard solution may be mixed together). The addition of 10 uL of the surrogate spiking solution to 5 gm of sediment/ soil is equivalent to 50 ug/kg of each surrogate standard.

1.7.2.1.3 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in 1.7.2.1 into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.1 gm.

1.7.2.1.3.1 Immediately after weighing the sample weigh 5-10 g of the sediment into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

Percent moisture

$$\frac{\text{gm of sample} - \text{gm of dry sample}}{\text{gm of sample}} \times 100 = \% \text{ moisture}$$

1.7.2.1.4 Add the spiked reagent water to the purge device and connect the device to the purge and trap system. NOTE: Steps 1.7.2.1.2 - 1.7.2.1.3, prior to the attachment of the purge device, must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

1.7.2.1.5 Heat the sample to 40°C ± 1°C and purge the sample for 12 ± 0.1 minutes.

1.7.2.1.6 Proceed with the analysis as outlined in 1.7.1.10 - 1.7.1.13. Use 5 mL of the same reagent water as the reagent blank.

1.7.2.1.7 For low level sediment/soils add 10 uL of the matrix spike solution (1.4.7) to the 5 mL of water (1.7.2.1.2). The concentration for a 5 gram sample would be equivalent to 50 ug/kg of each matrix spike standard.

1.7.2.2 Medium Level Method

The medium level method is based on extracting the sediment/soil sample with methanol. An aliquot of the methanol extract is added to reagent water containing the surrogate and internal standards. This is purged at ambient temperature. All samples with an X Factor >1.0 should be analyzed by the medium level method. If saturated peaks occurred or would occur when a 1 gram sample was analyzed, the medium level method must be used.

1.7.2.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatent liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4 gm (wet weight) into a tared 15 mL vial. Use a top loading balance. Note a nd record the actual weight to the nearest 0.1 gm. Determine the percent moisture as in 1.7.2.1.3.1.

1.7.2.2.2 Quickly add 9.0 mL of methanol, then 1.0 mL of the surrogate spiking solution to the vial. Cap and shake for 2 minutes. NOTE: Steps 1.7.2.2.1 and 1.7.2.2.2 must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

1.7.2.2.3 Pipette for storage approximately 1 mL of extract to a GC vial using a disposable pipet. The remainder may be disposed of. Transfer approximately 1 mL of the reagent methanol to a GC vial for use as the method blank for each case or set of 20 samples, whichever is greater. These extracts may be stored in the dark at 4°C prior to analysis.

The addition of a 100 uL aliquot of each of these extracts in paragraph 1.7.2.2.6 will give a concentration equivalent to 6,200 ug/kg of each surrogate standard.

1.7.2.2.4 The GC/MS system should be set up as in 1.7.1.2 - 1.7.1.4. This should be done prior to the addition of the methanol extract to reagent water.

1.7.2.2.5 The following table can be used to determine the volume of methanol extract to add to the 5 mL of reagent water for analysis. If the Hexadecane screen procedure was followed use the X factor (Option B) or the estimated concentration (Option A) to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low level analysis to determine the appropriate volume. If the sample was submitted as a medium level sample, start with 100 uL. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of linear range of the curve.

X Factor	Estimated Concentration Range[1/]	Take this Volume of Methanol Extract[2/]
	ug/kg	uL
0.25 - 5.0	500 - 10,000	100
0.5 - 10.0	1000 - 20,000	50
2.5 - 50.0	5000 - 100,000	10
12.5 - 250	25,000 - 500,000	100 of 1/50 dilution[3/]

Calculate appropriate dilution factor for concentrations exceeding the table.

1/ Actual concentration ranges could be 10 to 20 times higher than this if the compounds are halogenated and the estimates are from GC/FID.

2/ The volume of methanol added to the 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 uL added to the syringe.

3/ Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

1.7.2.2.6 Remove the plunger from a 5 mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5 mL to allow volume for the addition of sample and standards. Add 10 uL of the internal standard solution. Also add the volume of methanol extract determined in 1.7.2.2.5 and a volume of methanol solvent to total 100 uL (excluding methanol in standards).

1.7.2.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the water/methanol sample into the purging chamber.

1.7.2.2.8 Proceed with the analysis as outlined in 1.7.1.9 - 1.7.1.13. Analyze all reagent blanks on the same instrument as the samples. The standards should also contain 100 uL of methanol to simulate the sample conditions.

1.7.2.2.9 For a matrix spike in the medium level sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution (1.4.6), and 1.0 mL of matrix spike solution (1.4.7) in paragraph 1.7.2.2.2. This results in a 6,200 ug/kg concentration of each matrix spike standard when added to a 4 gm sample. Add a 100 uL aliquot of this extract to 5 mL of water for purging (as per paragraph 1.7.2.2.6).

1.8 Qualitative Analysis

1.8.1 The target compounds listed in the Hazardous Substances List (HSL), Exhibit C, shall be identified by an analyst competent in the interpretation of mass spectra (see Bidder Pre-Award Laboratory Evaluation Criteria) by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: (1) elution of the sample component at the same GC relative retention time as the standard component, and (2) correspondence of the sample component and standard component mass spectra.

1.8.1.1 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

1.8.1.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the contractor's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, only if the contractor's GC/MS meets the daily turning requirements for BFB or DFTPP. These standard spectra may be obtained from the run used to obtain reference RRTs.

1.8.1.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

(1) All ions present in the standard mass spectra at a relative intensity greater than 10 % (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

(2) The relative intensities of ions specified in (1) must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 30 and 70 percent).

(3) Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. In Task III. the verification process should favor false negatives.

1.8.2 A library search shall be executed for Non-HSL sample components for the purpose of tentative identification. For this purpose, the most recent available version of the EPA/NIH Mass Spectral Library shall be used. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

1.8.2.1 Up to 10 substances of greatest apparent concentration not listed in Exhibit C for the purgeable organic fraction shall be tentatively identified via a forward search of the EPA/NIH mass spectral library. (Substances with responses less than 10% of the internal standard are not required to be searched in this fashion). Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

1.8.2.2 Guidelines for making tentative identification: (1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50 percent of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

(3) Molecular ions present in reference spectrum should be present in sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. Data system library reduction programs can sometimes create these discrepancies.

1.8.2.3 If in the opinion of the mass spectral specialist, no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e. unknown aromatic, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

1.9 Quantitative Analysis

1.9.1 HSL components identified shall be quantified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte. The

EICP area of the characteristic ions of analytes listed in Tables 2 and 3 are used. The response factor (RF) from the daily standard analysis is used to calculate the concentration in the sample. Use the response factor as determined in paragraph 1.5.3.3 and the following equations:

Water (low and medium level)

$$\text{Concentration} \quad \text{ug/L} = \frac{(A_x)(I_s)}{(A_{is})(RF)(V_o)}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard from Exhibit E.

I_s = Amount of internal standard added in nanograms (ng)

V_o = Volume of water purged in milliliters (mL) (take into account any dilutions)

Sediment/Soil (medium level)

$$\text{Concentration} \quad \text{ug/kg} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RF)(V_i)(W_s)(D)}$$

Sediment/Soil (low level)

$$\text{Concentration} \quad \text{ug/kg} = \frac{(A_x)(I_s)}{(A_{is})(RF)(W_s)(D)}$$

(Dry weight basis)

Where:

A_x, I_s, A_{is} = same as for water, above

V_t = Volume of total extract (uL) (use 10,000 uL or a factor of this when dilutions are made)

V_i = Volume of extract added (uL) for purging

$D = \frac{100 - \%\ \text{moisture}}{100}$

W_s = Weight of sample extracted (gm) or purged

1.9.2 An estimated concentration for Non-HSL components tentatively identified shall be quantified by the internal standard method. For quantification, the nearest internal standard free of interfereces shall be used.

1.9.2.1 The formula for calculating concentrations is the same as in paragraph 1.9.1. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. A response factor (RF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds as well as those identified as unknowns.

1.9.2.2 Xylenes (o,m, & p - isomers) are to be reported as total Xylenes. Since o- and p-Xylene overlap, the Xylenes must be quantitated versus m-Xylene. The concentration of all Xylene isomers must be added together to give the total.

1.9.3 Calculate surrogate standard recovery on all samples, blanks and spikes. Determine if recovery is within limits and report on appropriate form.

1.9.3.1 Calculation for surrogate recovery.

$$\text{Percent Surrogate Recovery} = \frac{Q_d}{Q_a} \times 100\%$$

where: Q_d = quantity determined by analysis

Q_a = quantity added to sample

1.9.3.2 If recovery is not within limits, the following is required:

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the sample data if any of the above checks reveal a problem.
- Reanalyze the sample if none of the above are a problem.
- Report the data from both analyses along with the surrogate data from both.

Table 2

Characteristic Ions for Surrogate and

Internal Standards for Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
SURROGATE STANDARDS		
4-Bromofluorobenzene	95	174, 176
1,2-Dichloroethane d-4	65	102
Toluene d-8	98	70, 100
INTERNAL STANDARDS		
Bromochloromethane	128	49, 130, 51
1,4-Difluorobenzene	114	63, 88
Chlorobenzene d-5	117	82, 119

Table 3
Characteristic Ions for Volatile HSL Compounds

Parameter	Primary Ion*	Secondary Ion(s)
Chloromethane	50	52
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Methylene chloride	84	49, 51, 86
Acetone	43	58
Carbon disulfide	76	78
1,1-Dichloroethene	96	61, 98
1,1-Dichloroethane	63	65, 83, 85, 98, 100
trans-1,2-Dichloroethene	96	61, 98
Chloroform	83	85
1,2-Dichloroethane	62	64, 100, 98
2-Butanone	72	57
1,1,1-Trichloroethane	97	99, 117, 119
Carbon tetrachloride	117	119, 121
Vinyl acetate	43	86
Bromodichloromethane	83	85, 129
1,1,2,2-Tetrachloroethane	83	85, 131, 133, 166
1,2-Dichloropropane	63	65, 114
trans-1,3-Dichloropropene	75	77
Trichloroethene	130	95, 97, 132
Dibromochloromethane	129	208, 206
1,1,2-Trichloroethane	97	83, 85, 99, 132, 134
Benzene	78	-
cis-1,3-Dichloropropene	75	77
2-Chloroethyl vinyl ether	63	65, 106
Bromoform	173	171, 175, 250, 252, 254, 256
2-Hexanone	43	58, 57, 100
4-Methyl-2-pentanone	43	58, 100
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
Chlorobenzene	112	114
Ethyl benzene	106	91
Styrene	104	78, 103
Total xylenes	106	91

* The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.

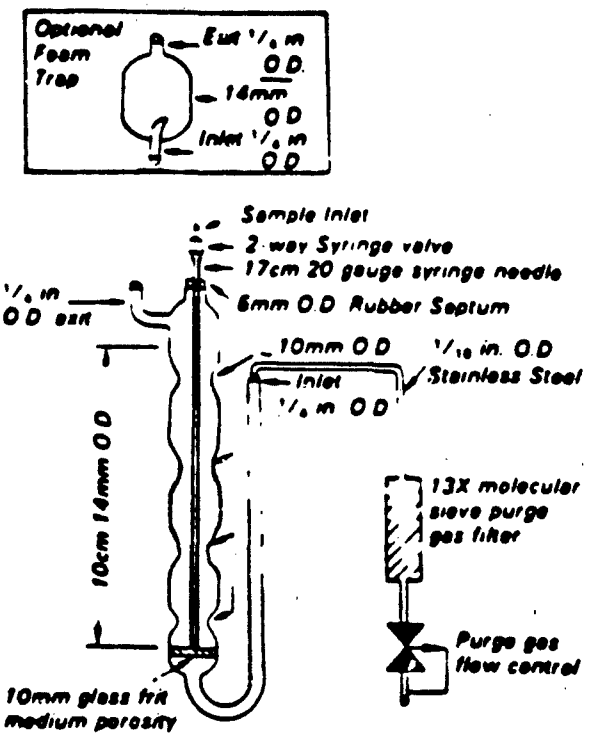

Figure 1. Purging device

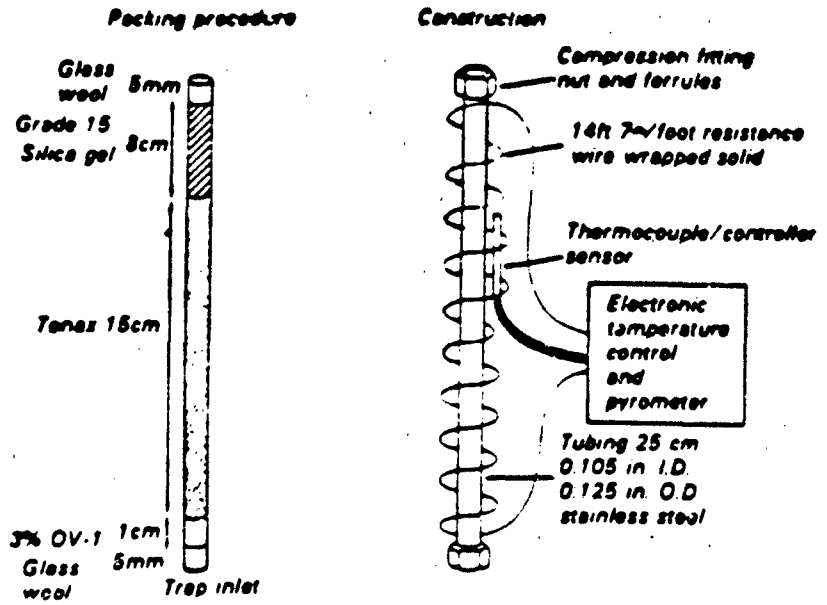

Figure 2. Trap packings and construction to include desorb capability

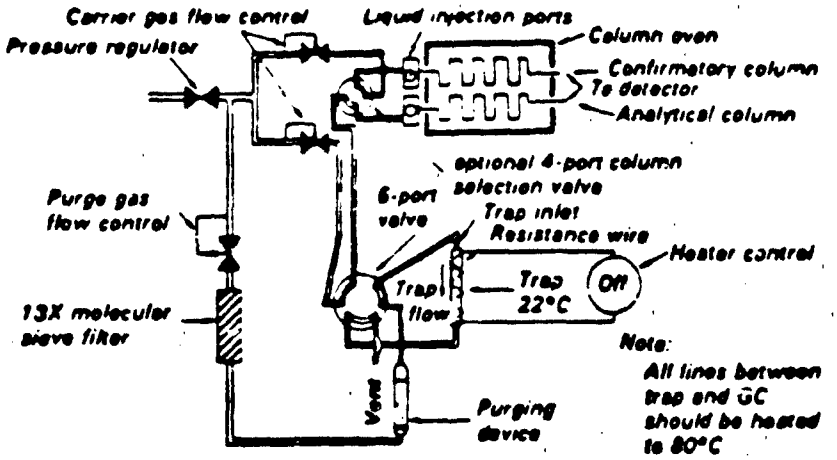

Figure 3 *Schematic of purge and trap device — purge mode*

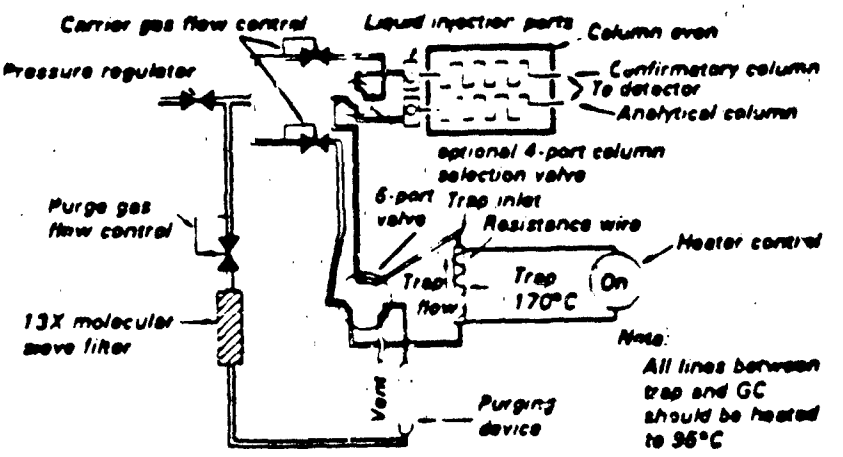

Figure 4. *Schematic of purge and trap device — desorb mode*

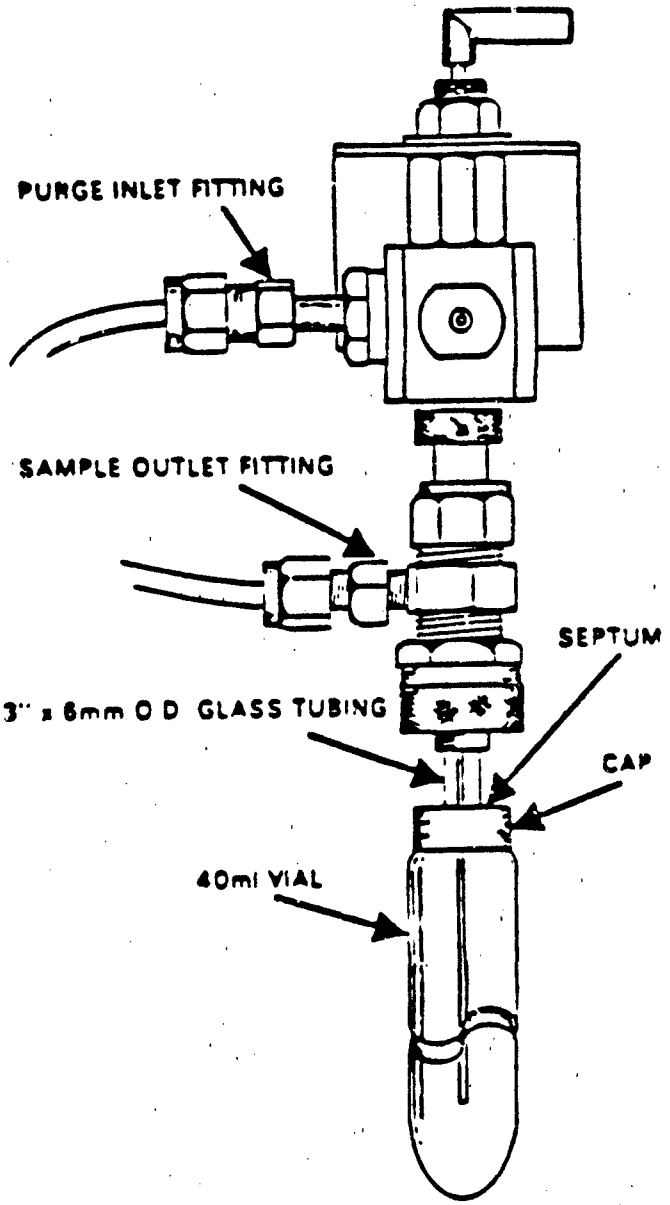

Figure 5. Low Soils Impinger

WESTON

STANDARD METHOD 209 G

6060A

209 G. Volatile and Fixed Matter in Nonfiltrable Residue and in Solid and Semisolid Samples

1. General Discussion

This method is applicable to the determination of total residue on evaporation and its fixed and volatile fractions in such solid and semisolid samples as river and lake sediments, sludges separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

The determination of both total and volatile residue in these materials is subject to negative error due to loss of ammonium carbonate [$(NH_4)_2CO_3$] and volatile organic matter while drying. Although this is true also for wastewater, the effect tends to be more pronounced with sediments, and especially with sludges and sludge cakes.

The mass of organic matter recovered from sludge and sediment requires a longer ignition time than that specified for residue from wastewaters, effluents, or polluted waters. Carefully observe specified ignition time and temperature to control losses of volatile inorganic salts.

Make all weighings quickly because wet samples tend to lose weight by evaporation. After drying or ignition, residues often are very hygroscopic and rapidly absorb moisture from the air.

2. Apparatus

See Sections 209A.2 and 209B.2.

3. Procedure

a. Solid and semisolid samples:

1) Total residue and moisture—

a) Preparation of evaporating dish—Ignite a clean evaporating dish at 550 ± 50 C for 1 hr in a muffle furnace. Cool in a desiccator, weigh, and store in a desiccator until ready for use.

b) Fluid samples—If the sample contains enough moisture to flow more or less readily, stir to homogenize, place 25 to 50 g in a prepared evaporating dish, and weigh to the nearest 10 mg. Evaporate to dryness on a water bath, dry at 103 C for 1 hr, cool in an individual desiccator containing fresh desiccant, and weigh.

c) Solid samples—If the sample consists of discrete pieces of solid material (dewatered sludge, for example), take cores from each piece with a No. 7 cork borer or pulverize the entire sample coarsely on a clean surface by hand, using rubber gloves. Place 2 to 50 g in a prepared evaporating dish and weigh to the nearest 10 mg. Place in an oven at 103 C overnight. Cool in an individual desiccator containing fresh desiccant and weigh. Prolonged heating may result in a loss of volatile organic matter and $(NH_4)_2CO_3$, but it usually is necessary to dry samples thoroughly.

2) Volatile residue—Determine volatile residue, including organic matter and volatile inorganic salts, on the total residue

obtained in 1) above. Avoid loss of solids by decrepitation by placing dish in a cool muffle furnace, heating furnace to 550 C, and igniting for 60 min. (First ignite samples containing large amounts of organic matter over a gas burner and under an exhaust hood in the presence of adequate air to lessen losses due to reducing conditions and to avoid odors in the laboratory.) Cool in a desiccator and reweigh. Report results as fixed residue (percent ash) and volatile residue.

b. Nonfiltrable residue (suspended matter):

1) Preparation of glass-fiber filter—Place a glass-fiber filter in a membrane filter holder, Hirsch funnel, or Buchner funnel, with wrinkled surface of filter facing upward. Apply vacuum to the assembled apparatus to seat filter. With vacuum applied, wash filter with three successive 20-mL portions of distilled water. After the water has filtered through, disconnect vacuum, remove filter, transfer to an aluminum or stainless steel planchet as a support, and dry in an oven at 103 C for 1 hr (30 min in a mechanical convection oven). If volatile matter is not to be determined, cool filter in a desiccator to balance temperature and weigh. If volatile matter is to be determined, transfer filter to a muffle furnace and ignite at 550 C for 15 min. Remove filter from furnace, place in a desiccator until cooled to balance temperature, and weigh.

2) Treatment of sample—Except for samples that contain high concentrations of filtrable matter, or that filter very slowly, select a sample volume ≥14 mL/cm² filter area.

Place prepared filter in membrane filter holder, Hirsch funnel, or Buchner funnel, with wrinkled surface upward. With vacuum applied, wet filter with distilled water to seat it against holder or funnel. Measure well-mixed sample with a wide-tip pipet or graduated cylinder. Filter sample through filter using suction. Leaving suction on, wash apparatus three times with 10-mL portions of distilled water, allowing complete drainage between washings. Discontinue suction, remove filter and dry to constant weight (see 209B.3c) at 103 C for 1 hr in an oven (30 min in a mechanical convection oven). After drying, cool filter in a desiccator to balance temperature and weigh.

3) Filtration with Gooch crucibles—Alternatively, use glass-fiber filters of 2.2 or 2.4 cm diam with Gooch crucibles and follow the procedure in Section 209D.3b.

4) Ignition—Ignite filter with its nonfiltrable residue (total suspended matter) for 15 min at 550 ± 50 C, transfer to a desiccator, cool to balance temperature, and weigh.

4. Calculation

a. Solid and semisolid samples:

$$\% \text{ total residue} = \frac{A \times 100}{B}$$

$$\% \text{ volatile residue} = \frac{(A - C) \times 100}{A}$$

$$\% \text{ fixed residue} = \frac{C \times 100}{A}$$

b. Nonfiltrable residue (suspended matter):

$$\text{mg nonfiltrable volatile residue/L} = \frac{(D - E) \times 1{,}000}{\text{sample volume, mL}}$$

$$\text{mg nonfiltrable fixed residue/L} = \frac{C \times 1{,}000}{\text{sample volume, mL}}$$

where:

A = weight of dried solids, mg,
B = weight of wet sample, mg,
C = weight of ash, mg,
D = weight of residue before ignition, mg, and
E = weight of residue after ignition, mg.

5. Precision and Accuracy

See Section 209D.5.

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APPENDIX D

SUPPLEMENTAL DATA

TABLE D-1. SOIL TEMPERATURE (°F)

Time	Test Run 1	Test Run 2	Test Run 3	Test Run 4
0 (1150)	77	75	68	57
5	71	85	59	52
10	71	90	59	52
15	74	91	62	53
20	77	90	64	56
25	77	90	74	59
30	79	88	78	62
35	79	88	100	65
40	80	87	108	68
45	81	87	119	72
50	81	88	119	75
55	81	88	118	81
60	81	89	123	85
65	81	90	126	89
70	81	91	---	---
75	81	91	---	---
80	82	90	109	100
85	83	91	112	104
90	87	90	116	108
95	90	91	118	111
100	95	92	123	113
105	98	92	127	113
110	99	92	129	114
115	103	91	125	115
120	106	91	126	116
125	*	91	125	117
130	*	91	125	117
135	*	91	128	117
140	---	92	127	118
145	---	92	---	---
150	141	92	---	---
155	141	91	114	118
160	142	91	118	109
165	141	91	121	112
170	141	92	123	112

*Thermocouple popped out of soil, temperature measured represented air temperature in the unit.

--- Not measured (sampling soil).

TABLE D-1. (CONTINUED)

Time	Test Run 1	Test Run 2	Test Run 3	Test Run 4
175	143	92	123	115
180	144	91	124	116
185	143	91	123	118
190	143	92	121	117
195	143	92	120	118
200	143	92	121	120
205	---	92	121	122
210	144	93	123	121
215	---	92	124	121
220	143	91	---	---
225	---	91	123	---
230	140	91	128	113
235		92	123	118
240		93	128	118
245		93	128	115
250			128	116
255			128	116
260			130	120
265			130	121
270			128	120
275			129	121
280			128	121
285			128	122

--- Not measured (sampling soil).

TABLE D-2. TOTAL VOC CONCENTRATION IN OUTLET AIR STREAM (PPM/VOLUME AS BENZENE)

Time	Test Run 1	Test Run 2	Test Run 3	Test Run 4
0	---	---	---	---
5	---	---	---	---
10	---	---	---	---
15	---	---	---	---
20	21	12	7	94
25	20	11	6	93
30	19	11	6	90
35	18	10	6	88
40	18	10	5	87
45	18	10	5	85
50	18	10	4	83
55	18	9	4	81
60	18	9	4	80
65	18	9	4	76
70	17	9	---	---
75	18	9	---	---
80	18	8	4	73
85	18	8	3	67
90	17	8	3	66
95	18	8	3	70
100	17	8	3	68
105	18	7	3	66
110	17	7	3	65
115	16	7	2	62
120	15	6	2	62
125	15	6	3	68
130	14	5	2	72
135	14	5	3	72
140	12	4	3	72
145	---	4	3	---
150	9	3	3	---

--- Not measured (sampling soil).

TABLE D-2. (CONTINUED)

Time	Test Run 1	Test Run 2	Test Run 3	Test Run 4
155	7	3	2	71
160	5	3	2	61
165	5	2	2	60
170	5	2	2	60
175	4	2	2	60
180	5	2	2	60
185	5	2	2	62
190	5	2	2	62
195	4	2	1	61
200	4	2	2	62
205	---	2	1	62
210	3	2	1	62
215	---	2	1	63
220	3	1	1	---
225	---	1	1	---
230	2	1	1	60
235	---	1	1	59
240	2	1	1	60
245	---		1	61
250	---		1	64
255	---		1	65
260	2		1	65
265			1	63
270			1	60
275			1	56
280			1	53
285			1	51

--- Not measured (sampling soil).

TABLE D-3. AIR TEMPERATURES (°F)

Time	Test run 1		Test run 2		Test run 3		Test run 4	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
0	90	94	140	33	89	83	89	64
5	109	86	138	84	113	77	111	63
10	134	84	139	89	137	75	123	64
15	145	84	140	90	139	77	128	64
20	152	84	140	91	144	77	132	67
25	156	85	141	91	148	77	133	79
30	160	86	140	90	151	77	135	68
35	162	87	141	90	152	77	136	69
40	165	87	140	90	152	78	138	72
45	164	88	140	91	151	77	137	73
50	166	89	142	92	152	78	138	73
55	166	89	142	92	154	80	137	73
60	167	90	143	94	152	79	139	76
65	168	90	143	96	151	78	---	---
70	169	91	143	98	---	---	---	---
75	169	92	143	98	130	77	130	73
80	168	91	143	100	144	78	133	73
85	168	91	143	102	147	81	137	74
90	169	91	143	103	148	81	137	74
95	167	92	144	104	152	82	137	75
100	168	98	143	108	155	83	138	77
105	167	100	143	108	156	83	138	77
110	166	100	143	110	153	84	140	79
115	166	100	145	111	152	88	140	80
120	167	100	145	111	153	90	140	82
125	167	100	145	113	155	92	138	80
130	167	100	142	113	152	94	140	81
135	168	104	143	114	153	97	140	82
140	168	106	143	115	154	99	---	---
145	---	---	143	116	---	---	---	---
150	169	108	144	116	135	91	128	80

--- Not measured (sampling soil).

TABLE D-3. (CONTINUED)

Time	Test run 1		Test run 2		Test run 3		Test run 4	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
155	170	114	146	118	146	91	133	83
160	165	117	147	118	150	91	137	83
165	170	120	149	119	148	90	137	83
170	171	120	148	119	149	90	140	84
175	173	121	148	120	149	92	140	88
180	175	125	149	120	149	92	140	90
185	172	124	148	120	149	94	140	90
190	169	123	147	120	150	97	143	92
195	170	123	147	120	150	97	144	93
200	170	122	148	120	150	96	143	94
205	---	---	147	120	152	97	143	96
210	167	124	147	120	152	97	144	97
215	---	---	147	120	151	98	---	---
220	168	124	147	120	---	---	---	---
225	---	---	147	120	147	94	135	88
230	166	122	147	120	152	94	139	90
235			147	120	154	96	141	90
240			148	120	154	100	143	92
245			146	121	155	100	144	93
250					155	100	144	94
255					155	102	146	95
260					156	104	146	95
270					156	104	147	97
275					157	104	148	98
280					156	104	145	99
285					154	104	145	100

--- Not measured (sampling soil).